# CANE SUGAR

AND

# ITS MANUFACTURE

H. C. PRINSEN GEERLIGS

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BY

H. C. PRINSEN GEERLIGS,

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GENERAL

# PREFACE.

THE aim of the present work is to compile in one book everything that is known about the chemistry and the technology of the sugar cane and cane sugar manufacture.

In doing this, the discussion of technicalities relating to machinery has been carefully avoided, because this subject is already sufficiently dealt with in Noel Deerr's work, "Sugar and the Sugar Cane." Only those points are mentioned here which are indispensable for the proper understanding of the chemical and technical questions investigated in this work.

Although the author has chiefly made use of his own investigations and researches carried on during his 17 years' continuous stay in Java at the head of the West Java Sugar Experiment Station, he has also gladly and gratefully referred to the results of work done by his colleagues in other parts of the world. In every case, where reference is made to the work of others, this has been acknowledged in a footnote.

The enumeration of the chemical and physical characteristics of the three kinds of sugar is chiefly borrowed from Prof. Dr. E. O. von Lippmann's excellent handbook, "Die Chemie der Zuckerarten."

In agreement with that distinguished scientist, the author has used in his book Fischer's nomenclature for the different kinds of sugar, because the terms dextrose and levulose are fast becoming obsolete in scientific literature and are apt to cause confusion.

In the present work the different terms have the following signification:—

SUGAR: The commercial product.

SUCROSE: The chemical body, the principal constituent of the commercial product.

GLUCOSE: The chemical body, also called dextrose.

FRUCTOSE: The chemical body, also called levulose.

INVERT SUGAR: The mixture of exactly equal proportions of glucose and fructose.

REDUCING SUGAR: Mixtures of uneven proportions of glucose and fructose.

The author has pleasure in acknowledging his great indebtedness to two gentlemen, Mr. James P. Ogilvie, Associate Editor of the *International Sugar Journal*, and Mr. T. H. P. Heriot—especially the latter—for a painstaking and laborious revision of the English text.

H. C. PRINSEN GEERLIGS.

AMSTERDAM, May, 1909.

192537

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# FIRST PART.

# THE RAW MATERIAL.

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CHAPTER I.

# THE CONSTITUENTS OF THE SUGAR CANE.

## I.—Cane Sugar or Sucrose.

Sugar is found in a great many plants, dissolved in the sap. Although it may be considered as one of the most universal constituents of plants, only a few of them contain it in such a degree and so sparingly mixed with other bodies as to allow of its profitable extraction. Such are: the sugar cane, the beetroot, the sorghum, the coco, date, palmyra and nipah palms, and the maple tree; then bees extract it from the flowers of many plants, but in this case it is soon converted into invert sugar.

**Occurrence in Nature.**

Sucrose appears in the form of anhydrous, bright, monoclinic crystals, having a pure, sweet taste, and possessing a specific gravity of 1·581 at 15°C. Large crystals, candy for instance, which have crystallized out slowly from hot solutions, are formed by the combination of parallelic disposed lamellae, which generally include parts of the mother-liquor in their interstices. Such a crystal can be split like a diamond along its axis by a sharp, heavy blow with a hammer. The occluded mother-liquor can cause coloration of the candy crystals; moreover, owing to its presence, candy, which externally is perfectly dry, on pulverization often yields a moist powder. When a broken crystal of sucrose is placed in a supersaturated solution of the same substance, and allowed to grow there, the damaged portion will gradually become healed, until the original form of the crystal is restored; after that crystalline growth proceeds regularly, provided it is allowed to do so in every direction, as, for example, when the crystal is suspended by means of a thread in a supersaturated sucrose solution, which is thickened with isinglass or agar-agar, or when it is kept in regular and constant motion in a supersaturated solution.

**Crystalline Form.**

Sucrose crystallizes from its aqueous solution in crystals, which are larger in proportion as the liquid in which they are formed contains a less amount of bodies other than sucrose and as the crystallization is slower. Very rapid crystallization or crystallization from syrups containing a large amount of foreign substances is apt to yield small sucrose crystals.

Strongly supersaturated sucrose solutions when agitated will suddenly deposit very minute so-called "mealy" crystals, whilst steady agitation of moderately supersaturated sucrose solutions causes the crystals to grow as slowly and continuously as when at rest. In this case they become even more regular, because, owing to the constant motion, they are free in every direction and are able to move throughout the whole of the supersaturated liquid, which gives them a more regular crystalline form than if they had remained deposited on the bottom of the crystallization vessel.

Sucrose, even when it is in a pure state, sometimes crystallizes in an abnormal form from solutions containing certain foreign bodies, such as raffinose, zinc salts, some calcium salts, or products resulting from the overheating of sugar. The influence of these substances is, however, not always perceptible, as sucrose will sometimes crystallize in its usual form from solutions containing a large proportion of raffinose. The abnormality of the crystalline form of sucrose consists in the stretching of one of the axes of the crystal, which causes it to assume the shape of a needle. This becomes very obvious when the crystals combine into bundles.

**Solubility in Water.**

Sucrose is freely soluble in water and its solubility increases as the temperature rises. The values for the solubility of sucrose at different temperatures are recorded in the following table compiled by Herzfeld*:—

| Temperature. | Per cent. of Sucrose. | Temperature. | Per cent. of Sucrose. | Temperature. | Per cent. of Sucrose. | Temperature. | Per cent. of Sucrose. | Temperature. | Per cent. of Sucrose. |
|---|---|---|---|---|---|---|---|---|---|
| °C. | | °C. | | °C. | | °C. | | °C. | |
| 0 | 64·18 | 14 | 66·18 | 28 | 68·37 | 42 | 70·78 | 56 | 73·39 |
| 1 | 64·30 | 15 | 66·33 | 29 | 68·53 | 43 | 70·96 | 57 | 73·58 |
| 2 | 64·45 | 16 | 66·48 | 30 | 68·70 | 44 | 71·14 | 58 | 73·78 |
| 3 | 64·59 | 17 | 66·63 | 31 | 68·87 | 45 | 71·32 | 59 | 73·98 |
| 4 | 64·73 | 18 | 66·78 | 32 | 69·04 | 46 | 71·50 | 60 | 74·18 |
| 5 | 64·87 | 19 | 66·93 | 33 | 69·21 | 47 | 71·68 | 61 | 74·38 |
| 6 | 65·01 | 20 | 67·09 | 34 | 69·38 | 48 | 71·87 | 62 | 74·58 |
| 7 | 65·15 | 21 | 67·25 | 35 | 69·55 | 49 | 72·06 | 63 | 74·78 |
| 8 | 65·29 | 22 | 67·41 | 36 | 69·72 | 50 | 72·25 | 64 | 74·98 |
| 9 | 65·43 | 23 | 67·57 | 37 | 69·89 | 51 | 72·44 | 65 | 75·18 |
| 10 | 65·58 | 24 | 67·73 | 38 | 70·06 | 52 | 72·63 | 66 | 75·38 |
| 11 | 65·73 | 25 | 67·89 | 39 | 70·24 | 53 | 72·82 | 67 | 75·59 |
| 12 | 65·88 | 26 | 68·05 | 40 | 70·42 | 54 | 73·01 | 68 | 75·80 |
| 13 | 66·03 | 27 | 68·21 | 41 | 70·60 | 55 | 73·20 | 69 | 76·01 |

* *Zeitsch. Rübenzuckerind.*, 1892, 181.

| Temperature. | Per cent. of Sucrose. | Temperature. | Per cent. of Sucrose. | Temperature. | Per cent. of Sucrose. | Temperature. | Per cent. of Sucrose. | Temperature. | Per cent. of Sucrose. |
|---|---|---|---|---|---|---|---|---|---|
| °C. | | °C. | | °C. | | °C. | | °C. | |
| 70 | 76·22 | 77 | 77·70 | 84 | 79·24 | 91 | 80·84 | 98 | 82·49 |
| 71 | 76·43 | 78 | 77·92 | 85 | 79·46 | 92 | 81·07 | 99 | 82·73 |
| 72 | 76·64 | 79 | 78·14 | 86 | 76·69 | 93 | 81·30 | 100 | 82·79 |
| 73 | 76·85 | 80 | 78·36 | 87 | 79·92 | 94 | 81·53 | — | — |
| 74 | 77·06 | 81 | 78·58 | 88 | 80·15 | 95 | 81·77 | — | — |
| 75 | 77·27 | 82 | 78·80 | 89 | 80·38 | 96 | 82·01 | — | — |
| 76 | 77·48 | 83 | 79·02 | 90 | 80·61 | 97 | 82·25 | — | — |

General formula for the solubility: $y = 64{\cdot}1835 + 0{\cdot}13477\,x + 0{\cdot}0005307\,x^2$. $y$ = per cent. of sugar, and $x$ = temp. in degrees C.

**Contraction on being dissolved in Water.**

When sucrose is dissolved in water a considerable contraction takes place; this reaches its maximum at 56 per cent., as may be seen from the following table:—

| Sucrose. Grms. | Water. Grms. | Volume. c.c. | Sucrose. Grms. | Water. Grms. | Volume. c.c. |
|---|---|---|---|---|---|
| 0 | 100 | 100·0000 | 55·0 | 45·0 | 99·0059 |
| 10 | 90 | 99·7218 | 55·9 | 44·1 | 99·0055 |
| 20 | 80 | 99·4732 | 56·0 | 44·0 | 99·0042 |
| 30 | 70 | 99·2822 | 56·1 | 43·9 | 99·0055 |
| 40 | 60 | 99·1103 | 57·0 | 43·0 | 99·0059 |
| 50 | 50 | 99·0219 | — | — | — |
| 60 | 40 | 99·0121 | — | — | — |
| 70 | 30 | 99·0921 | — | — | — |
| 80 | 20 | 99·2756 | — | — | — |
| 90 | 10 | 99·5745 | — | — | — |
| 100 | 0 | 100·0000 | — | — | — |

* E. O. von Lippmann, *Chemie der Zuckerarten*, II., 1081.

**Sp. Gr. of Solutions of Sucrose in Water.**

According to Brix tables the specific gravity of sucrose solutions is at a temperature of 15° C. as follows :—

| Per Cent. | Specific Gravity. | Per Cent. | Specific Gravity. | Per Cent. | Specific Gravity. |
|---|---|---|---|---|---|
| 1 | 1·00388 | 23 | 1·09686 | 45 | 1·20565 |
| 2 | 1·00779 | 24 | 1·10145 | 46 | 1·21100 |
| 3 | 1·01173 | 25 | 1·10607 | 47 | 1·21639 |
| 4 | 1·01570 | 26 | 1·11072 | 48 | 1·22182 |
| 5 | 1·01970 | 27 | 1·11541 | 49 | 1·22728 |
| 6 | 1·02373 | 28 | 1·12013 | 50 | 1·23278 |
| 7 | 1·02779 | 29 | 1·12488 | 51 | 1·23832 |
| 8 | 1·03187 | 30 | 1·12967 | 52 | 1·24390 |
| 9 | 1·03599 | 31 | 1·13449 | 53 | 1·24951 |
| 10 | 1·04014 | 32 | 1·13934 | 54 | 1·25571 |
| 11 | 1·04431 | 33 | 1·14423 | 55 | 1·26086 |
| 12 | 1·04852 | 34 | 1·14915 | 56 | 1·26658 |
| 13 | 1·05276 | 35 | 1·15411 | 57 | 1·27235 |
| 14 | 1·05703 | 36 | 1·15911 | 58 | 1·27816 |
| 15 | 1·06133 | 37 | 1·16413 | 59 | 1·28400 |
| 16 | 1·06566 | 38 | 1·16920 | 60 | 1·28989 |
| 17 | 1·07002 | 39 | 1·17430 | 61 | 1·29581 |
| 18 | 1·07441 | 40 | 1·17934 | 62 | 1·30177 |
| 19 | 1·07884 | 41 | 1·18460 | 63 | 1·30777 |
| 20 | 1·08329 | 42 | 1·18981 | 64 | 1·31381 |
| 21 | 1·08778 | 43 | 1·19505 | 65 | 1·31989 |
| 22 | 1·09231 | 44 | 1·20033 | 66 | 1·32601 |

Gerlach calculated the following formula for the specific gravity of $x$ per cent. of sucrose dissolved at 17·5° C. :—

$$y = 1 + 0{\cdot}00386571327\,x + 0{\cdot}00001414091916\,x^2 + 0{\cdot}0000000328794657176\,x^3$$

from which values Scheibler computed the following interpolation formulæ :—

$t = 0°$, $y = 1 + 0{\cdot}003976844\,x + 0{\cdot}0000142764\,x^2 + 0{\cdot}000000029120\,x^3$
$t = 10°$, $y = 1 + 0{\cdot}003915138\,x + 0{\cdot}0000139524\,x^2 + 0{\cdot}000000032728\,x^3$
$t = 15°$, $y = 1 + 0{\cdot}003884496\,x + 0{\cdot}0000139399\,x^2 + 0{\cdot}000000033806\,x^3$
$t = 20°$, $y = 1 + 0{\cdot}003844136\,x + 0{\cdot}0000144092\,x^2 + 0{\cdot}000000030912\,x^3$
$t = 30°$, $y = 1 + 0{\cdot}003796428\,x + 0{\cdot}0000145456\,x^2 + 0{\cdot}000000030664\,x^3$
$t = 40°$, $y = 1 + 0{\cdot}003764028\,x + 0{\cdot}0000143700\,x^2 + 0{\cdot}000000035192\,x^3$
$t = 50°$, $y = 1 + 0{\cdot}003722992\,x + 0{\cdot}0000148080\,x^2 + 0{\cdot}000000032440\,x^3$
$t = 60°$, $y = 1 + 0{\cdot}003683112\,x + 0{\cdot}0000155904\,x^2 + 0{\cdot}000000026368\,x^3$

Although yet other formulæ have been suggested, those mentioned here are quite sufficient for ordinary purposes.

**Influence of Temperature on the Sp. Gr. of Sucrose Solutions.**

The specific gravity of sucrose solutions diminishes as the temperature rises, because the rise in temperature increases their volume. Gerlach determined the following values for the specific gravity of solutions of different concentrations, at various temperatures, in which water at 17·5° C. = 1.

| ° C. | 0 per cent. Sucrose. | 15 per cent. Sucrose. | 30 per cent. Sucrose. | 45 per cent. Sucrose. | 60 per cent. Sucrose. | 75 per cent. Sucrose. |
|---|---|---|---|---|---|---|
| 0 | 1·0007 | 1·0636 | 1·1337 | 1·2113 | 1·2972 | 1·3916 |
| 20 | 0·9996 | 1·0606 | 1·1288 | 1·2046 | 1·2889 | 1·3822 |
| 40 | 0·9942 | 1·0504 | 1·1212 | 1·1958 | 1·2794 | 1·3722 |
| 60 | 0·9857 | 1·0448 | 1·0881 | 1·1851 | 1·2683 | 1·3610 |
| 80 | 0·9745 | 1·0336 | 1·0768 | 1·1729 | 1·2562 | 1·3488 |
| 100 | 0·9621 | 1·0202 | 1·0634 | 1·1597 | 1·2424 | 1·3356 |

At the suggestion of Stammer, Sachs calculated from these figures the following table of corrections for the Brix hydrometer at temperatures different from 17·5° C. which has come into universal use in sugar house laboratories:—

| Temp. of the Liquid. | Degrees Brix. | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | 0 | 5 | 10 | 15 | 20 | 25 | 30 | 35 | 40 | 50 | 60 | 70 | 75 |
| | Subtract from the readings | | | | | | | | | | | | |
| 15 | 0·09 | 0·11 | 0·12 | 0·14 | 0·14 | 0·15 | 0·16 | 0·17 | 0·16 | 0·17 | 0·19 | 0·21 | 0·25 |
| 16 | 0·06 | 0 07 | 0·08 | 0·09 | 0·10 | 0·10 | 0·11 | 0·12 | 0·12 | 0·12 | 0·14 | 0·16 | 0·18 |
| 17 | 0·02 | 0·02 | 0·03 | 0·03 | 0·03 | 0·04 | 0·04 | 0·04 | 0·04 | 0·04 | 0·05 | 0·05 | 0·06 |
| | Add to the readings | | | | | | | | | | | | |
| 18 | 0·02 | 0·03 | 0·03 | 0·03 | 0·03 | 0·03 | 0·03 | 0·03 | 0·03 | 0·03 | 0·03 | 0·03 | 0·02 |
| 19 | 0·06 | 0·08 | 0·08 | 0·09 | 0·09 | 0·10 | 0·10 | 0·10 | 0·10 | 0·10 | 0·10 | 0·08 | 0·06 |
| 20 | 0·11 | 0·14 | 0·15 | 0·17 | 0·17 | 0·18 | 0·18 | 0·18 | 0·19 | 0·19 | 0·18 | 0·15 | 0·11 |
| 21 | 0·16 | 0 20 | 0·22 | 0·24 | 0·24 | 0·25 | 0·25 | 0·25 | 0·26 | 0·26 | 0·25 | 0·22 | 0·18 |
| 22 | 0·21 | 0·26 | 0·29 | 0·31 | 0·31 | 0·32 | 0·32 | 0·32 | 3·33 | 0·34 | 0·32 | 0·29 | 0·25 |
| 23 | 0·27 | 0·32 | 0·35 | 0·37 | 0·38 | 0·39 | 0·39 | 0·39 | 0·40 | 0·42 | 0·39 | 0·36 | 0·33 |
| 24 | 0·32 | 0·38 | 0·41 | 0·43 | 0·44 | 0·46 | 0·46 | 0·47 | 0·47 | 0·50 | 0·46 | 0 43 | 0·40 |
| 25 | 0·37 | 0·44 | 0·47 | 0·49 | 0·51 | 0·53 | 0·54 | 0·55 | 0·55 | 0·58 | 0·54 | 0·51 | 0·48 |
| 26 | 0·43 | 0·50 | 0·54 | 0·56 | 0·58 | 0·60 | 0·61 | 0·62 | 0·62 | 0·66 | 0·62 | 0·58 | 0·55 |
| 27 | 0·49 | 0·57 | 0·61 | 0·63 | 0·65 | 0·68 | 0·68 | 0·69 | 0·70 | 0·74 | 0·70 | 0·65 | 0·62 |
| 28 | 0·56 | 0·64 | 0·68 | 0·70 | 0·72 | 0·76 | 0·76 | 0·78 | 0 ·78 | 0·82 | 0·78 | 0·72 | 0·70 |
| 29 | 0·63 | 0·71 | 0·75 | 0·78 | 0·79 | 0·84 | 0·84 | 0·86 | 0·86 | 0·90 | 0·86 | 0·80 | 0·70 |

| Temp. of the Liquid. | Degrees Brix. | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | 0 | 5 | 10 | 15 | 20 | 25 | 30 | 35 | 40 | 50 | 60 | 70 | 75 |
| | Add to the readings | | | | | | | | | | | | |
| 30 | 0·70 | 0·78 | 0·82 | 0·87 | 0·87 | 0·92 | 0·92 | 0·94 | 0·94 | 0·98 | 0·94 | 0·88 | 0·88 |
| 35 | 1·10 | 1·17 | 1·22 | 1·24 | 1·30 | 1·32 | 1·33 | 1·35 | 1·36 | 1·39 | 1·34 | 1·27 | 1·25 |
| 40 | 1·50 | 1·61 | 1·67 | 1·71 | 1·73 | 1·79 | 1·79 | 1·80 | 1·82 | 1·83 | 1·78 | 1·69 | 1·65 |
| 50 | — | 2·65 | 2·71 | 2·74 | 2·78 | 2·80 | 2·80 | 2·80 | 2·80 | 2·79 | 2·70 | 2·56 | 2·51 |
| 60 | — | 3·87 | 3·88 | 3·88 | 3·88 | 3·88 | 3·88 | 3·88 | 3·90 | 3·82 | 3·70 | 3·43 | 3·41 |
| 70 | — | 5·17 | 5·18 | 5·20 | 5·14 | 5·13 | 5·10 | 5·08 | 5·06 | 4·90 | 4·72 | 4·47 | 4·35 |
| 80 | — | — | 6·62 | 6·59 | 6·54 | 6·46 | 6·38 | 6·30 | 6·26 | 6·06 | 5·82 | 5·50 | 5·33 |
| 90 | — | — | 8·26 | 8·16 | 8·06 | 7·97 | 7·83 | 7·71 | 7·58 | 7·30 | 6·96 | 6·58 | 6·37 |
| 100 | — | — | 10·01 | 9·87 | 9·72 | 9·56 | 9·39 | 9·21 | 9·03 | 8·64 | 8·22 | 7·76 | 7·42 |

**Boiling Point of Sucrose Solutions.**

According to Gerlach's researches, the boiling point of sucrose solutions of different concentration is at the ordinary atmospheric pressure :—

| Per cent. Sucrose | 10 | 20 | 30 | 40 | 50 | 60 | 70 | 80 | 90·8 |
|---|---|---|---|---|---|---|---|---|---|
| Temperature .. | 100·4 | 100·6 | 101·0 | 101·5 | 102·0 | 103·0 | 106·5 | 112·0 | 130·0° C. |

**Solubility of Sucrose in Alcohol and other Liquids.**

Sucrose is very sparingly soluble in absolute alcohol, viz., only one part sucrose dissolves in 80 parts of absolute alcohol at the boiling point; but dilute alcohol dissolves it much more readily, and this in proportion as the alcohol is more dilute and the temperature higher, as is shown in the following table compiled by Pellet.* The first column represents the c.c. of absolute alcohol present in 100 c.c. of the solution and the other columns the grms. of sucrose dissolved in 100 c.c. of the mixture at temperatures up to 40° C.

| c.c. Absolute Alcohol. | Grms. sucrose in 100 c.c. at | | | | |
|---|---|---|---|---|---|
| | 0° | 10° | 20° | 30° | 40° |
| 0 | 85·5 | 87·3 | 88·3 | 91·8 | 94·2 |
| 10 | 80·7 | 82·5 | 85·0 | 87·0 | 90·0 |
| 20 | 74·2 | 76·5 | 79·0 | 82·0 | 84·0 |
| 30 | 65·5 | 68·0 | 71·0 | 74·0 | 77·5 |
| 40 | 56·7 | 59·0 | 62·0 | 65·5 | 69·0 |
| 50 | 45·9 | 48·5 | 52·0 | 55·6 | 60·0 |
| 60 | 32·9 | 35·5 | 39·0 | 42·5 | 47·5 |
| 70 | 18·2 | 20·0 | 22·5 | 26·0 | 31·0 |
| 80 | 6·4 | 7·2 | 8·1 | 10·1 | 14·0 |
| 90 | 0·7 | 0·8 | 1·1 | 1·6 | 2·3 |
| 97·4 | 0 08 | 0·15 | 0·25 | 0·36 | 0·50 |

* *Bull. Assoc. Chim. Sucr. et Dist.*, **15**, 631.

In each case less sucrose is dissolved in a mixture of alcohol and water than could be dissolved at the same temperature in the amount of water present in the mixture, if it had not been mixed with alcohol.

Sucrose also dissolves in dilute methyl alcohol, but not in chloroform, ether and similar solvents, and it is practically insoluble in anhydrous glycerine.

**Action of the Electric Current on Sucrose Solutions.**

In aqueous solutions, sucrose is a non-conductor of electricity, but after a prolonged action of the current a slight conductivity is apparent. This, however, is not due to the sucrose itself but either to impurities in the water used for solution or from decomposition products of the sucrose, as the electric current, provided it is of a sufficient power, will attack sucrose, inverting it with formation of acids.

**Rotatory Power.**

Aqueous sucrose solutions deflect the plane of the polarized light ray to the right with a specific rotatory power which is only insignificantly influenced by the concentration of the solution. According to Landholt, the specific rotatory power of sucrose is, for concentrations coming into consideration in sugar house work (which never exceed 24 per cent.*), expressed in arc degrees :—

$$\alpha \frac{20}{D} = 66{\cdot}435 + 0{\cdot}00870\, c - 0{\cdot}000235\, c^2$$

in which formula $c$ stands for concentration.

**Influence of Concentration on Rotatory Power.**

By this formula, the figures for the rotation of sucrose for the different concentrations are as follows :—

| c. | Rotation. | c. | Rotation. | c. | Rotation. |
|---|---|---|---|---|---|
| 1 | 66·443 | 25 | 66·506 | 50 | 66·283 |
| 5 | 66·473 | 30 | 66·485 | 55 | 66·203 |
| 10 | 66·499 | 35 | 66·452 | 60 | 66·111 |
| 15 | 66·513 | 40 | 66·407 | 65 | 66·007 |
| 20 | 66·515 | 45 | 66.351 | | |

At low concentrations the rotatory power increases considerably and amounts, according to Nasini and Villavechia, to :—

| $c$ | 1·2588 | 1·2378 | 1·2083 | 1·0129 | 0·8255 | 0·6631 | 0·5985 | 0·5880 | 0·3350 |
|---|---|---|---|---|---|---|---|---|---|
| $\alpha \frac{20}{D}$ | 66·604 | 66·716 | 66·855 | 67·096 | 67·250 | 67·370 | 67·562 | 67·983 | 68·241 |

At higher concentrations, the differences in the rotatory power are so small that within the limits of concentration met with in all sugar house polarization work they do not affect the results, whilst at lower concentrations, where they are more considerable, the figure which they influence is itself so small, that even there the difference is insignificant. In the well-known Schmitz

* $\frac{26{\cdot}048}{1{\cdot}1} = 23{\cdot}68$

tables for the calculation of the sucrose content from the polarimeter readings, the influence of the concentration is taken into consideration.

**Influence of Temperatures.**

The rotatory power of sucrose diminishes as the temperature rises to an extent, which for the simple rotatory power itself—quite apart from changes in the instrument brought about by differences of the temperature,—can according to Schönrock's calculation be expressed by the formula:—

$$a\,\frac{t}{D} = a\,\frac{20}{D} - a\,\frac{20}{D} \times 0{\cdot}000217\ (t^\circ - 20).$$

In practical working this figure is also influenced by the alterations in the rotatory power of the quartz wedges of the polarimeter, by the expansion of the polarization tubes, &c., which cause the correction for the Ventzke scale with glass polarimeter tubes to become

$$\text{readings at } t^\circ = \text{readings at } 20^\circ\ [1 - 0{\cdot}0003\ (t^\circ - 20)]$$

which means that for every degree Ventzke the rotation is found to be 0·0003° V. too low for every degree Centigrade above 20° C. (the temperature at which the polarimeter is standardized), and the same amount too high for every degree Centigrade below that temperature of 20° C.

**Influence of Foreign Bodies on the Rotatory Power.**

When sucrose is dissolved in water containing alcohol, its rotatory power is slightly higher than when pure water is used; thus, for a concentration of 10 per cent. of sucrose in a mixture of 1 part of water and 3 parts of alcohol it becomes at 20° C. 66·287 instead of 66·499. Alkalies, alkali carbonates and alkaline earths reduce the specific rotation of sucrose, probably owing to the formation of saccharates, as neutralization with acid restores the original rotation.

The reduction of the rotatory power by alkaline substances is much more considerable in concentrated than in dilute solutions, and amounts, according to Pellet, for the alkalies, their carbonates, and the alkaline earths, to the following values:—

At concentrations $c = 5{\cdot}4$ and $c = 17{\cdot}3$, 1 grm. of the undermentioned substances marks the rotation of the stated quantities of sucrose in grammes.

| | $c = 5{\cdot}4$. | $c = 17{\cdot}3$. |
|---|---|---|
| 1 grm. of caustic potash .. .. .. | 0·170 .. | 0·500 |
| 1 ,, ,, soda .. .. .... | 0·140 .. | 0·450 |
| 1 ,, potassium carbonate .. .. | 0·044 .. | 0·065 |
| 1 ,, sodium carbonate .. .... | 0·040 .. | 0·132 |
| 1 ,, ammonia .. .. .. .. | 0·073 .. | 0·085 |
| 1 ,, ammonium carbonate .... | 0·040 .. | 0·067 |
| 1 ,, lime .. .. .. .. .. | 0·700 .. | 1·000 |
| 1 ,, baryta.. .. .. .. .... | 0·190 .. | 0·430 |

**Influence of Basic Lead Acetate on Rotatory Power.**

Basic lead acetate, though also an alkaline substance, does not affect the rotation of sucrose to such an extent.

Bates and Blake* stated the influence of basic lead acetate solution of 1·25 specific gravity on the rotation of a solution of 26·048 grms. sucrose in 100 c.c. to be in degrees Ventzke:—

| c.c. Basic Lead Acetate Solution. | Difference. | c.c. Basic Lead Acetate Solution. | Difference. |
|---|---|---|---|
| 0·5 | 0·09 | 10·0 | +0·19 |
| 1·0 | 0·13 | 12·0 | +0·29 |
| 1·5 | 0·10 | 15·0 | +0·29 |
| 2·0 | 0·13 | 18·0 | +0·45 |
| 2·5 | 0·06 | 20·0 | +0·45 |
| 3·0 | 0·08 | 25·0 | +0·58 |
| 4·0 | 0·06 | 30·0 | +0·62 |
| 5·0 | 0·03 | 35·0 | +0·77 |
| 6·0 | 0·00 | 40·0 | +0·77 |
| 7·0 | +0·05 | 63·0 | +0·95 |
| 8·0 | +0·09 | | |

The addition of small quantities of basic acetate solution therefore causes a progressive reduction in the polarimeter reading; with larger quantities of the reagent the reduction becomes less; the solution to which 6 c.c. of basic lead solution has been added gives the same polarimeter reading as one without lead, while still larger quantities cause a progressive increase of the rotation. As in sugar-house work the excess of basic lead never goes beyond a few drops, we may, in ordinary practice, safely neglect the influence of this reagent on the polariscopic reading.

**Neutral Salts.**

Alkali chlorides, sulphates, and phosphates, as well as the chlorides of the alkaline earths, reduce the rotation of sucrose solutions, as is shown by the following table of Bodenbender and Steffens. †

| | Sucrose. | Salt. | Water. | Polarization. | Difference. |
|---|---|---|---|---|---|
| Potassium chloride .... | 5 | 1 | 94 | 4·987 | 0·013 |
| | 10 | 2 | 88 | 9·856 | 0·144 |
| | 20 | 4 | 76 | 19·869 | 0·131 |
| Sodium chloride .. .. | 5 | 1 | 94 | 4·969 | 0·131 |
| | 10 | 2 | 88 | 9·853 | 0·147 |
| | 20 | 4 | 76 | 19·586 | 0·414 |

* *Jl. Amer. Chem. Soc.*, 1907, 286. † *Zeitsch. Rübenzuckerind.*, **31**, 808.

| | Sucrose. | Salt. | Water. | Polarization. | Difference. |
|---|---|---|---|---|---|
| Barium chloride.. .... | 5 | 1 | 94 | 4·952 | 0·048 |
| | 10 | 2 | 88 | 9·944 | 0·056 |
| | 20 | 4 | 76 | 19·402 | 0·598 |
| Magnesium sulphate .. | 5 | 1 | 94 | 4·995 | 0·005 |
| | 10 | 2 | 88 | 9·890 | 0·109 |
| | 20 | 4 | 76 | 19·880 | 0·120 |
| Sodium phosphate .... | 5 | 1 | 94 | 4·958 | 0·042 |
| | 10 | 2 | 88 | 9·933 | 0·067 |
| | 20 | 4 | 76 | 19·689 | 0·311 |

In the presence of small amounts of inorganic salts such as are met with in ordinary sugar-house routine work the influence of the salt is imperceptible. With proportions of 4 parts of the inorganic salt to 20 parts of sucrose the product itself is so impure that the absolutely certain determination of the sucrose by polarization is rendered impossible both by the large volume of the precipitate occasioned by the clarification and also by the dark coloration of the liquid. Besides, clarification with basic lead acetate converts all the salts into acetates, the action of which is less than that of the original salts, so that in practical working the effect of these inorganic salts on the rotatory power of sucrose may safely be neglected.

**Refractive Index of Sucrose Solutions.**

According to Stolle* the refractive index of sucrose solutions for mixed light at 17·5° C. is, taking water = 1·3310

| Concentration. | Specific Gravity. | Refractive Index. | Concentration. | Specific Gravity. | Refractive Index. |
|---|---|---|---|---|---|
| 0·9979 | 1·00241 | 1·33465 | 25·0120 | 1·09420 | 1·36891 |
| 4·0073 | 1·01406 | 1·33889 | 35·0219 | 1·13194 | 1·38306 |
| 12·0052 | 1·04484 | 1·35044 | 45·8381 | 1·17246 | 1·39873 |
| 17 9385 | 1·06736 | 1·35891 | 55·0266 | 1·20651 | 1·41150 |

Other investigators have found higher values than the above. Stolle has determined the refractive indices of glucose and fructose solutions of different concentrations, and his results will be found further on in this work.

* *Zeitsch. Rübenzuckerind.*, 51, 469.

Tolman and Smith* determined the refractive indices of sucrose solutions at 20° C. to be as follows :—

| Per cent. Sucrose. | Refractive Index. | Per cent. Sucrose. | Refractive Index. | Per cent. Sucrose. | Refractive Index. | Per cent. Sucrose. | Refractive Index. |
|---|---|---|---|---|---|---|---|
| 1 | 1·3343 | 24 | 1·3705 | 47 | 1·4137 | 70 | 1·4653 |
| 2 | 1·3357 | 25 | 1·3722 | 48 | 1·4158 | 71 | 1·4677 |
| 3 | 1·3372 | 26 | 1·3739 | 49 | 1·4179 | 72 | 1·4701 |
| 4 | 1·3387 | 27 | 1·3756 | 50 | 1.4200 | 73 | 1·4726 |
| 5 | 1·3402 | 28 | 1·3774 | 51 | 1·4221 | 74 | 1·4751 |
| 6 | 1·3417 | 29 | 1·3792 | 52 | 1·4242 | 75 | 1 4776 |
| 7 | 1·3432 | 30 | 1·3810 | 53 | 1·4263 | 76 | 1·4801 |
| 8 | 1·3447 | 31 | 1·3828 | 54 | 1·4284 | 77 | 1·4826 |
| 9 | 1·3462 | 32 | 1·3847 | 55 | 1·4306 | 78 | 1·4851 |
| 10 | 1·3477 | 33 | 1·3865 | 56 | 1·4328 | 79 | 1·4877 |
| 11 | 1·3492 | 34 | 1·3883 | 57 | 1·4351 | 80 | 1·4903 |
| 12 | 1·3508 | 35 | 1·3902 | 58 | 1·4373 | 81 | 1·4929 |
| 13 | 1·3524 | 36 | 1·3921 | 59 | 1·4396 | 82 | 1·4955 |
| 14 | 1·3539 | 37 | 1·3940 | 60 | 1·4419 | 83 | 1·4981 |
| 15 | 1·3555 | 38 | 1·3959 | 61 | 1·4442 | 84 | 1·5007 |
| 16 | 1·3572 | 39 | 1·3978 | 62 | 1·4465 | 85 | 1·5034 |
| 17 | 1·3588 | 40 | 1·3997 | 63 | 1·4488 | 86 | 1·5061 |
| 18 | 1·3604 | 41 | 1·4017 | 64 | 1·4511 | 87 | 1·5088 |
| 19 | 1·3621 | 42 | 1·4036 | 65 | 1·4534 | 88 | 1·5115 |
| 20 | 1·3637 | 43 | 1·4056 | 66 | 1·4557 | 89 | 1·5142 |
| 21 | 1·3654 | 44 | 1·4076 | 67 | 1·4581 | 90 | 1·5170 |
| 22 | 1·3671 | 45 | 1·4096 | 68 | 1·4605 | — | — |
| 23 | 1·3688 | 46 | 1·4117 | 69 | 1·4629 | — | — |

The same workers have investigated the necessary correction for the influence of temperature on the refractive index, and summarize their results as follows :—

* *Jl. Amer. Chem. Soc.*, 1906, 1480.

| Per cent. Sucrose. | Refractive Index at 20° C. | Refractive Index at 30° C. | Difference. | Correction for 10° C. | |
|---|---|---|---|---|---|
| | | | | In per cent. Sucrose. | In degrees Brix. |
| 2·18 | 1·3358 | 1·3348 | 0·0010 | 0·66 | 0·64 |
| 7·43 | 1·3438 | 1·3428 | 0·0010 | 0·66 | 0·67 |
| 15·82 | 1·3569 | 1·3557 | 0·0012 | 0·70 | 0·70 |
| 51·71 | 1·4236 | 1·4219 | 0·0017 | 0·81 | 0·79 |
| 62·52 | 1·4477 | 1·4459 | 0·0018 | 0·78 | 0·76 |

The author has determined the refractive index of pure sucrose solutions at 28° C. (standard temperature for tropical work), and also the influence of temperature on the readings, the results of which investigations are recorded in the two tables given below:—*

| Refractive Index. | Per cent. Sucrose. | Decimals. | |
|---|---|---|---|
| 1·3335 | 1 | 0·0001=0·05 | 0·0010=0·75 |
| 1·3349 | 2 | 0·0002=0·1 | 0·0011=0·8 |
| 1·3364 | 3 | 0·0003=0·2 | 0·0012=0·8 |
| 1·3379 | 4 | 0·0004=0·25 | 0·0013=0·85 |
| 1·3394 | 5 | 0·0005=0·3 | 0·0014=0·9 |
| 1·3409 | 6 | 0·0006=0·4 | 0·0015=1·0 |
| 1·3424 | 7 | 0·0007=0·5 | |
| 1·3439 | 8 | 0·0008=0·6 | |
| 1·3454 | 9 | 0·0009=0·7 | |
| 1·3469 | 10 | | |
| 1·3484 | 11 | 0·0001=0·05 | |
| 1·3500 | 12 | 0·0002=0·1 | |
| 1·3516 | 13 | 0·0003=0·2 | |
| 1·3530 | 14 | 0·0004=0·25 | |
| 1·3546 | 15 | 0·0005=0·3 | |
| 1·3562 | 16 | 0·0006=0·4 | |
| 1·3578 | 17 | 0·0007=0·45 | |
| 1·3594 | 18 | 0·0008=0·5 | |
| 1·3611 | 19 | 0·0009=0·6 | |
| 1·3627 | 20 | 0·0010=0·65 | |
| 1·3644 | 21 | 0·0011=0·7 | |

| Refractive Index. | Per cent. Sucrose. | Decimals. | |
|---|---|---|---|
| 1·3661 | 22 | 0·0012=0·75 | |
| 1·3678 | 23 | 0·0013=0·8 | |
| 1·3695 | 24 | 0·0014=0·85 | |
| 1·3712 | 25 | 0·0015=0·9 | |
| 1·3729 | 26 | 0·0016=0·95 | |
| 1·3746 | 27 | 0·0001=0·05 | 0·0012=0·6 |
| 1·3764 | 28 | 0·0002=0·1 | 0·0013=0·65 |
| 1·3782 | 29 | 0·0003=0·15 | 0·0014=0·7 |
| 1·3800 | 30 | 0·0004=0·2 | 0·0015=0·75 |
| 1·3818 | 31 | 0·0005=0·25 | 0·0016=0·8 |
| 1·3836 | 32 | 0·0006=0·3 | 0·0017=0·85 |
| 1·3854 | 33 | 0·0007=0·35 | 0·0018=0·9 |
| 1·3872 | 34 | 0·0008=0·4 | 0·0019=0·95 |
| 1·3890 | 35 | 0·0009=0·45 | 0·0020=1·0 |
| 1·3909 | 36 | 0·0010=0·5 | 0·0021=1·0 |
| 1·3928 | 37 | 0·0011=0·55 | |
| 1·3947 | 38 | | |
| 1·3966 | 39 | | |
| 1·3984 | 40 | | |
| 1·4003 | 41 | | |

* *International Sugar Journal*, 1908, 68.

| Refractive Index. | Per cent. Sucrose. | Decimals. | |
|---|---|---|---|
| 1·4023 | 42 | 0·0001=0·05 | 0·0012=0·6 |
| 1·4043 | 43 | 0·0002=0·1 | 0·0013=0·65 |
| 1·4063 | 44 | 0·0003=0·15 | 0·0014=0·7 |
| 1·4083 | 45 | 0·0004=0·2 | 0·0015=0·75 |
| 1·4104 | 46 | 0·0005=0·25 | 0·0016=0·8 |
| 1·4124 | 47 | 0·0006=0·3 | 0·0017=0·85 |
| 1·4145 | 48 | 0·0007=0·35 | 0·0018=0·9 |
| 1·4166 | 49 | 0·0008=0·4 | 0·0019=0·95 |
| 1·4186 | 50 | 0·0009=0·45 | 0·0020=1·0 |
| 1·4207 | 51 | 0·0010=0·5 | 0·0021=1·0 |
| 1·4228 | 52 | 0·0011=0·55 | |
| 1·4249 | 53 | | |
| 1·4270 | 54 | | |
| 1·4292 | 55 | 0·0001=0·05 | 0·0013=0·55 |
| 1·4314 | 56 | 0·0002=0·1 | 0·0014=0 6 |
| 1·4337 | 57 | 0·0003=0·1 | 0·0015=0·65 |
| 1·4359 | 58 | 0·0004=0·15 | 0·0016=0·7 |
| 1·4382 | 59 | 0·0005=0·2 | 0·0017=0·75 |
| 1·4405 | 60 | 0·0006=0·25 | 0.0018=0·8 |
| 1·4428 | 61 | 0·0007=0·3 | 0·0019=0·85 |
| 1·4451 | 62 | 0·0008=0·35 | 0·0020=0·9 |
| 1·4474 | 63 | 0·0009=0·4 | 0·0021=0·9 |
| 1·4497 | 64 | 0·0010=0·45 | 0·0022=0·95 |
| 1·4520 | 65 | 0·0011=0·5 | 0·0023=1·0 |
| 1·4543 | 66 | 0·0012=0·5 | 0·0024=1·0 |
| 1·4567 | 67 | | |
| 1·4591 | 68 | | |
| 1·4615 | 69 | | |
| 1·4639 | 70 | | |
| 1·4663 | 71 | | |
| 1·4687 | 72 | | |
| 1·4711 | 73 | 0·0001=0·0 | 0·0015=0·55 |
| 1·4736 | 74 | 0·0002=0·05 | 0·0016=0·6 |
| 1·4761 | 75 | 0·0003=0·1 | 0·0017=0·65 |
| 1·4786 | 76 | 0·0004=0·15 | 0·0018=0·65 |
| 1·4811 | 77 | 0·0005=0·2 | 0·0019=0·7 |
| 1·4836 | 78 | 0·0006=0·2 | 0·0020=0·75 |
| 1·4862 | 79 | 0·0007=0·25 | 0·0021=0·8 |
| 1·4888 | 80 | 0·0008=0·3 | 0·0022=0·8 |
| 1·4914 | 81 | 0·0009=0·35 | 0·0023=0·85 |
| 1·4940 | 82 | 0·0010=0 35 | 0·0024=0·9 |
| 1·4966 | 83 | 0·0011=0·4 | 0·0025=0·9 |
| 1·4992 | 84 | 0·0012=0·45 | 0·0026=0·95 |
| 1·5019 | 85 | 0 0013=0·5 | 0·0027=1·0 |
| 1·5046 | 86 | 0·0014=0·5 | 0·0028=1·0 |
| 1·5073 | 87 | | |
| 1·5100 | 88 | | |
| 1·5127 | 89 | | |
| 1·5155 | 90 | | |

## Table of Corrections for the Temperature.

| Temp. of the Prisms in °C. | Sucrose. | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | 0 | 5 | 10 | 15 | 20 | 25 | 30 | 40 | 50 | 60 | 70 | 80 | 90 |
| | Subtract | | | | | | | | | | | | |
| 20 | 0·53 | 0·54 | 0·55 | 0·56 | 0·57 | 0·58 | 0·60 | 0·62 | 0·64 | 0·62 | 0·61 | 0·60 | 0·58 |
| 21 | 0·46 | 0·47 | 0·48 | 0·49 | 0·50 | 0·51 | 0·52 | 0·54 | 0·56 | 0·54 | 0·53 | 0·52 | 0·50 |
| 22 | 0·40 | 0·41 | 0·42 | 0·42 | 0·43 | 0·44 | 0·45 | 0·47 | 0·48 | 0·47 | 0·46 | 0·45 | 0·44 |
| 23 | 0·33 | 0·33 | 0·34 | 0·35 | 0·36 | 0·37 | 0·38 | 0·39 | 0·40 | 0·39 | 0·38 | 0·38 | 0·38 |
| 24 | 0·26 | 0·26 | 0·27 | 0·28 | 0·28 | 0·29 | 0·30 | 0·31 | 0·32 | 0·31 | 0·31 | 0·30 | 0·30 |
| 25 | 0·20 | 0·20 | 0·21 | 0·21 | 0·22 | 0·22 | 0·23 | 0·23 | 0·24 | 0·23 | 0·23 | 0·23 | 0·22 |
| 26 | 0·12 | 0·12 | 0·13 | 0·14 | 0·14 | 0·14 | 0·15 | 0·15 | 0·16 | 0·16 | 0·16 | 0·15 | 0·14 |
| 27 | 0·07 | 0·07 | 0·07 | 0·07 | 0·07 | 0·07 | 0·08 | 0·08 | 0·08 | 0·08 | 0·08 | 0·08 | 0·07 |

| Temp. of the Prisms in °C. | Sucrose. Add 0 | 5 | 10 | 15 | 20 | 25 | 30 | 40 | 50 | 60 | 70 | 80 | 90 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 29 | 0·07 | 0·07 | 0·07 | 0·07 | 0·07 | 0·07 | 0·08 | 0·08 | 0·08 | 0·08 | 0·08 | 0·08 | 0·07 |
| 30 | 0·12 | 0·12 | 0·13 | 0·14 | 0·14 | 0·14 | 0·15 | 0·15 | 0·16 | 0·16 | 0·16 | 0·15 | 0·14 |
| 31 | 0·20 | 0·20 | 0·21 | 0·21 | 0·22 | 0·22 | 0·23 | 0·23 | 0·24 | 0·23 | 0·23 | 0·23 | 0·22 |
| 32 | 0·26 | 0·26 | 0·27 | 0·28 | 0·28 | 0·29 | 0·30 | 0·31 | 0·32 | 0·31 | 0·31 | 0·30 | 0·30 |
| 33 | 0·33 | 0·33 | 0·34 | 0·35 | 0·36 | 0·37 | 0·38 | 0·39 | 0 40 | 0·39 | 0·38 | 0·38 | 0·38 |
| 34 | 0·40 | 0·41 | 0·42 | 0·42 | 0·43 | 0·44 | 0·45 | 0·47 | 0·48 | 0 47 | 0 46 | 0·45 | 0·44 |
| 35 | 0·46 | 0·47 | 0·48 | 0·49 | 0·50 | 0·51 | 0·52 | 0·54 | 0·56 | 0·54 | 0·53 | 0·52 | 0·50 |

**Viscosity.**

The viscosity of sucrose solutions increases as the concentration rises and decreases as the temperature rises. This is shown by the following two tables, compiled by Claassen.* In these the number of seconds which 100 c.c. of a sucrose solution of different concentrations require to flow out from an Engler viscosimeter is recorded as rate of flow. V (viscosity) means the ratio of these figures calculated on 100, being the rate of flow of the saturated sucrose solution at the temperature of 30° C. of 219 parts of sucrose on 100 parts of water.

| Sucrose on 100 water. | Rate of flow. Time in seconds. | V = Viscosity. Saturated solution = 100. |
|---|---|---|
| 100 | 22 | 13 |
| 160 | 47 | 29 |
| 188 | 88 | 54 |
| 219 | 164 | 100 |
| 224 | 187 | 114 |
| 230 | 217 | 132 |
| 236 | 248 | 151 |
| 242 | 280 | 171 |
| 248 | 318 | 194 |
| 256 | 373 | 227 |
| 270 | 464 | 283 |

The second table shows the rate of flow of a sucrose solution saturated at 30° C. when examined at different temperatures.

* *Zeitsch. Rübenzuckerind.*, **48**, 535.

| Temperature. °C. | Rate of flow. Time in seconds. | Temperature. °C. | Rate of flow. Time in seconds. |
|---|---|---|---|
| 16 | 476 | 42 | 86 |
| 21 | 330 | 51 | 54 |
| 25 | 246 | 61 | 39 |
| 30 | 163 | 71 | 30 |
| 37 | 114 | | |

The third table gives the rate of flow of sucrose solutions of increasing concentrations at increasing temperatures, and shows that the decrease in viscosity by the rise in temperature is much more considerable than its increase by the higher concentration.

| Sucrose on 100 water. | Per cent. Water. | Per cent. Sucrose. | Temperature in °C. | Rate of flow. in seconds. |
|---|---|---|---|---|
| 201 | 33·3 | 66·7 | 17 | 180 |
| 206 | 32·7 | 67·3 | 22 | 161 |
| 229 | 30·4 | 69·6 | 35 | 110 |
| 273 | 26·8 | 73·2 | 55 | 63 |
| 294 | 25·4 | 74·6 | 63 | 55 |
| 323 | 23·6 | 76·4 | 71 | 51 |

The values for the viscosity of impure sucrose solutions, *e.g.*, molasses which generally contain as much foreign substance as sucrose, differ much from those given above, a point that will be dealt with later on in the chapter on molasses.

**Behaviour of Sucrose at high Temperature.**

Perfectly dry sucrose may be heated for a long time at temperatures exceeding 100° C., and even melted at 160° C., without undergoing any decomposition, but in presence of even traces of water it is decomposed at temperatures above 100° C.; this is shown by the dark colour it assumes, and the property it acquires of reducing Fehling's solution.

Sucrose, when heated for some time at its melting point, changes without any loss of weight into a mixture of glucose and fructosan (levulosan).

$$C_{12}H_{22}O_{11} = C_6H_{12}O_6 + C_6H_{10}O_5.$$

On heating sucrose at a still higher temperature it loses water, and leaves behind a dark coloured body called caramel; if the temperature is further

raised this is decomposed into gaseous products such as carbon dioxide, carbon monoxide, formic acid, acetone, &c., and a glossy coke which, on combustion in contact with air, burns entirely away and leaves no residue, provided the sugar used in the experiment was pure.

**Calorific Value.** On combustion in a bomb calorimeter, sucrose gives a calorific value of 3955·2 kg/kg calories or 7119·4 B.T.U.

**Decomposition of Sucrose Solutions on boiling.** On continuously heating sucrose solutions at their boiling point for a long time, the sucrose combines with water even at the ordinary atmospheric pressure, and gradually changes into a mixture of equal parts of glucose and fructose.

The material composing the vessel in which the solution is boiled exerts a considerable influence on the rapidity of the transformation; copper has the greatest accelerating action in this respect, then silver, next platinum, whilst with glass vessels the rate of hydrolysis is least of all, especially when a water-bath, and not a naked flame, is used as the source of heat. In such determinations inconvenience is caused by the formation of small quantities of acid, so that the values thereby found do not represent the amount of hydrolysis due to water alone. In order to remove this source of error, Herzfeld, in his researches on the decomposition of sucrose at high temperatures with prolonged action, rendered his solutions slightly alkaline at the commencement of the experiment. The results he obtained on heating sucrose solutions having an initial alkalinity of 0·01—0·05 per cent. are summarized in the following table; the solutions contained in metal vessels were heated in a water or oil bath at the temperature given in the first column of the table. The figures in the first table refer to the quantity of sucrose decomposed per hour per 100 parts of solution, and those in the second give the amount of sucrose decomposed during the same period of time per 100 parts of sucrose originally present.

| ° C. | 10 per cent. | 30 per cent. | 50 per cent. |
|---|---|---|---|
| 80 | 0·0044 | 0·0047 | 0·0100 |
| 90 | 0·0079 | 0·0087 | 0·0196 |
| 100 | 0·0114 | 0·0127 | 0·0292 |
| 110 | 0·0163 | 0·0167 | 0·0388 |
| 120 | 0·0282 | 0·0577 | 0·1399 |
| 130 | 0·2055 | 0·2600 | 0·5900 |
| 140 | 0·5100 | — | — |

| °C. | 10 per cent. | 15 per cent. | 20 per cent. | 25 per cent. | 30 per cent. | 35 per cent. | 40 per cent | 45 per cent. | 50 per cent. |
|---|---|---|---|---|---|---|---|---|---|
| 80 | 0·0444 | 0·0373 | 0·0301 | 0·0229 | 0·0157 | 0·0168 | 0·0179 | 0·0190 | 0·0200 |
| 90 | 0·0790 | 0·0667 | 0·0541 | 0·0418 | 0·0290 | 0·0317 | 0 0344 | 0·0371 | 0·0392 |
| 100 | 0·1140 | 0·0961 | 0·0781 | 0·0602 | 0·0423 | 0·0466 | 0·0508 | 0·0551 | 0·0584 |
| 110 | 0·1630 | 0·1362 | 0·1093 | 0·0825 | 0·0557 | 0·0612 | 0·0667 | 0·0721 | 0·0766 |
| 120 | 0·2823 | 0·2582 | 0·2341 | 0·2098 | 0·1857 | 0·2063 | 0·2669 | 0·2474 | 0·2678 |
| 130 | 2·0553 | 1·7582 | 1·4610 | 1·1638 | 0·8667 | 0·9451 | 1·0235 | 1·0119 | 1·1800 |
| 140 | 5·1000 | — | — | — | — | — | — | — | — |

Under a high pressure, coincident with a higher boiling point, this reaction takes place more rapidly, so that a solution is completely inverted after six hours' heating at 150° C., which if otherwise boiled at 100° C. would be totally transformed at the end of 24 hours.

Sucrose solutions, when boiled with live steam at 130° C., become very rapidly decomposed, and in the case of higher concentrations than those recorded in Herzfeld's tables, the loss by this means is undoubtedly considerable.

As a result of this decomposition besides glucose and fructose, carbon dioxide, formic acid, acetic acid, and many other more complex organic acids are formed. The temperature at which the decomposition reaches a maximum lies between 110° and 120° C., so that these temperatures ought to be avoided as much as possible in the course of manufacture.

The action of superheated steam also breaks up sucrose; at 160° C., a temperature which can occur in boilers, humic acid, carbonic acid, and formic acid are formed. At 280° sucrose is split up into carbonic acid and carbon with the formation of various empyreumatic products.

**Decomposition of Sucrose by Acids.** Dilute acids exert an hydrolysing power on sucrose, slowly at the ordinary temperature but quickly on heating. Thereby one molecule of sucrose combines with one molecule of water to form one molecule of fructose and one molecule of glucose.

$$C_{12}H_{22}O_{11} + H_2O = C_6H_{12}O_6 + C_6H_{12}O_6.$$

**Inversion.** The sugar thus transformed now deflects the plane of polarization to the left instead of to the right, hence the newly-formed body bears the name of invert sugar and the phenomenon itself that of inversion. During this transformation, 95 parts of sucrose combine with 5 parts of water to form 100 parts of invert sugar.

* *Zeitsch. Rübenzuckerind.*, **43**, 745.

**Inversion Velocity.** The velocity of the inversion depends on different laws, which have been formulated by Ostwald as follows:—

1. On inversion by a strong acid, sulphuric or hydrochloric acid, for instance, in every unit of time a constant part of the sucrose present is inverted; the value of the constant depends only on the acid used. Each acid has, therefore, its inversion constant, which is expressed by the formula $C \frac{1}{t} \times log \frac{A}{A - x}$, in which $A$ represents the quantity of sucrose initially present, and $x$ that of sucrose inverted, so that this constant is inversely proportional to the time in which the inversion is completed.

2. The velocity of inversion by equal quantities of the acid acting on different quantities of sucrose in the same volume is always the same. At the same concentration of the acid the constant is not influenced by the quantity of sucrose, so that even the most concentrated sucrose solutions can be inverted by minimum quantities of acid.

3. The action of the acid is in direct proportion to its concentration, though the inversion velocity of strong mineral acids increases more rapidly with progressive inversion than that of feeble ones.

4. The inversion velocity increases rapidly with the temperature.

**Inversion Constants of some Acids.** Ostwald determined the inversion constants of a number of acids by mixing 10 c.c. of a 40-50 per cent. sucrose solution at 25° C. with 10 c.c. of a normal solution of the acid, and ascertaining after a certain time the amount of invert sugar formed.

These constants, and the same values calculated on hydrochloric acid as a starting point with the figure 100, are shown in the table underneath.

| | Constant. | HCl = 100 | | Constant. | HCl. = 100 |
|---|---|---|---|---|---|
| Hydrobromic acid.. | 24·38 | 111·4 | Tartaric acid.. .. | 0·674 | 3·08 |
| Hydrochloric acid.. | 21·87 | 100·0 | Citric acid.. .... | 0·377 | 1·72 |
| Nitric acid .. .. | 21·87 | 100·0 | Formic acid.. .. | 0·335 | 1·53 |
| Sulphuric acid .... | 11·72 | 53·6 | Malic acid.. .... | 0·278 | 1·27 |
| Sulphurous acid .. | 6·63 | 30·4 | Lactic acid .. .. | 0·233 | 1·07 |
| Oxalic acid.. .... | 4·00 | 18·57 | Succinic acid .... | 0·119 | 0·545 |
| Phosphoric acid .. | 1·357 | 6·21 | Acetic acid .. .. | 0·088 | 0·400 |

It is true that these comparative figures are not of great practical value as they are based on the temperature of 25° C. and the inversion velocity of the acids rises very irregularly and unequally at higher temperatures. Thus it increases for sulphuric and sulphurous acid very rapidly between 30° and 40°, for oxalic

acid at 40°, for phosphoric acid between 40° and 50°, and for acetic acid between 70° and 80° C. The inversion constant of some acids is at 40° eight times, and at 55° even forty-eight times, as great as at 25° C.

The inversion constant of weak acids is measurable only at temperatures at which inversion by strong acids is so rapid that accurate determinations cannot be made, and it is for this reason that the compilation of trustworthy tables of comparison for higher temperatures is impossible. Hence Oswald's table is of little value in the sugar house, where but very slight amounts of weak acids are met with and high temperatures have to be considered. Moreover the presence of other bodies, such as glucose, exerts a notable influence on the inversion velocity of acids.

Besides acids, many salts, especially those of inorganic acids, possess the power of inverting sucrose solutions at the boiling point of their solutions, which power does not increase in proportion to their concentration. This must be ascribed to the fact that these salts occur in the solution in a partially dissociated state and that the part of the acid which is practically uncombined exerts its inverting power like a free acid. Such are, in fact, the salts of heavy metals as the sulphates of zinc, iron, and copper, which possess an acid reaction in solution.

**Inverting Power of Salts.**

When glucose, however, is also present, salts having a neutral reaction also show this property, and especially those which are very liable to dissociation, like ammonium salts or salts with feeble bases. Salts with a strong base or salts of organic acids (which even when free are but feeble hydrolysts), accordingly possess also a very slight inverting power or none at all, or can even neutralize by their mere presence the action of the other salts. Calcium carbonate too will prevent this action, so that we may safely conclude that the glucose acts here as a feeble acid, which liberates extremely small quantities of acid from the salts. In the case of the base and the acid of the salt being both strong, the glucose cannot exert much influence, and the dissociation as well as the inversion are insignificant; in the case of one of the two being feeble the dissociation is much greater; if the acid is strong, the inversion becomes appreciable; but if on the contrary the acid is weak, then it is impossible for it to act as a hydrolyst and no inversion can take place. In the simultaneous presence of salts having strong and weak acids, the latter are liberated and cannot invert; for the same reason presence of calcium carbonate prevents inversion by the action of the small amount of liberated acid, as it combines with the acid and thus neutralizes it.

As in cane juice the organic acids occur largely, and since therefore in neutralized juices and masse cuites the amount of organic salts is large in proportion to the inorganic ones, and further because the percentage of reducing sugars is, as far as first products go, very low when compared with that of the sucrose, there will be very little danger of inversion by neutral

salts in the practice of cane sugar manufacture from the milling to the after-products stage. But in the after-products and molasses, which generally possess an acid reaction and in which the reducing sugars have become concentrated, inversion at high temperatures is often observed; this is doubtless largely due to the presence of free acids, but not altogether, for it is possible that the action of inorganic salts may also account for it in some degree.

**Action of Concentrated Acids.**

Mineral acids of high concentration decompose sucrose. Gaseous hydrochloric acid transforms it into a mixture of ulmic acid and caramel; concentrated hydrochloric acid chars it; whilst a sucrose solution when boiled with dilute hydrochloric acid yields a series of decomposition products, which have been investigated by Berthelot and André. The principal products of this reaction are formic acid, levulinic acid, and humic acid, along with small quantities of carbonic acid, carbon monoxide, and furfural. Our own researches in this province will be dealt with in the discussion on glucose and fructose. Glucose forms chiefly levulinic acid, whilst humic acid is the principal decomposition product of fructose.

**Action of Alkalis and Alkaline Earths.**

Alkaline bodies, such as potash, soda, lime, &c., only attack sucrose solutions when they are heated with them in a concentrated state. On boiling a sucrose solution with a small percentage of potash or soda, it remains unchanged, but with higher concentrations of the alkali the sucrose becomes decomposed with the formation of formic acid, lactic acid, and humic bodies. Lime also attacks sucrose very slowly and one may even introduce small pieces of quicklime into a 10 per cent. sucrose solution and there allow them to slake without fear of decomposition occurring; but on boiling a sucrose solution with lime for a long time, the sugar is attacked, and yields acetic and lactic acids which of course remain in solution as lime salts.

**Saccharates.**

Moderately strong concentrations of solutions of alkalis and alkaline earths combine with sucrose and form soluble alkaline compounds called saccharates, which require just as much acid for their neutralization as the quantity of base in the compound.

**Alkali Saccharates.**

The formula of such a saccharate is, for instance, $C_{12}H_{21}O_{11}K$; after addition of carbonic acid to the solution of a saccharate it breaks up into sucrose and the alkali carbonate, but in aqueous solutions, even when very dilute, it is stable and cannot be decomposed by dialysis.

**Calcium Saccharate.**

The alkaline earths form a whole series of saccharates, of which some are soluble and others insoluble, and which, owing to their property of being readily decomposed by carbonic acid, may render good service in the separation of sucrose from molasses or from impure solutions in general.

The monobasic calcium saccharate $C_{12}H_{22}O_{11}CaO$ is soluble in water but by boiling its solution it is transformed into tribasic saccharate and sucrose.

$$3\ C_{12}H_{22}O_{11}CaO = C_{12}H_{22}O_{11}\ 3\ CaO + 2\ C_{12}H_{22}O_{11}.$$

The bisaccharate is also soluble in water and its solutions are likewise transformed into tribasic saccharate and sucrose on boiling.

On passing carbonic acid gas into a solution of calcium saccharate it is at first entirely absorbed but soon the mass becomes viscous and gelatinous and absorbs the carbonic acid much less readily. Finally, when the gas has been completely absorbed the liquid becomes thin again, calcium carbonate is precipitated and glucose is found in solution. The gelatinous character of the first-formed compound of carbonic acid and calcium saccharate $C_{12}H_{22}O_{11}3CaCO_3 2Ca(OH)_2$ causes the difficult absorption of the carbonic acid, which penetrates in large bubbles through the viscous liquid and for the greater part passes out unabsorbed; but when later on the liquid becomes fluid again by the gradual decomposition of the compound referred to above, the rate of the carbonic acid absorption is restored, and finally the saccharate becomes quantitatively split up into calcium carbonate and sucrose.

**Strontium Saccharate.**

Strontium bisaccharate is formed by dissolving strontium hydrate in a boiling 15 per cent. sucrose solution; the separation of the saccharate sets in as soon as more that two molecules of strontium hydrate are added to each molecule of sucrose present, and on the addition of further quantities of strontium hydrate almost all of the sucrose is precipitated as saccharate when about three molecules of strontium hydrate are present. The insolubility of strontium saccharate in alcohol is utilized in the separation of small amounts of sucrose from plant extracts.

To this end the plants are extracted with hot 90 per cent. alcohol, and the extract is filtered. To the hot filtrate so much strontium hydrate solution is added, that for each part of sucrose more than three parts of the hydrate are present. The mixture is boiled for half an hour, filtered, and the precipitate washed with hot alcohol. The liquid is boiled again with strontium hydrate for half an hour, and filtered through a hot water funnel. The precipitates are pressed as dry as possible, suspended in water, and decomposed by a current of carbonic acid. Strontium carbonate is thrown down, whilst the filtered sucrose solution can be evaporated and allowed to crystallize.

**Ferric Saccharate.**

Ferric saccharate is formed by allowing iron to dissolve slowly in sucrose solution in presence of oxygen. The ferric saccharates of the Pharmacopœia are not true saccharates, since all of them contain alkali; a part of their iron content is, however, present in the form of saccharates. Ferric saccharates may be obtained by precipitating ferric chloride dissolved in a sucrose solution with sodium hydrate, filtering the precipitate off, washing it, and suspending it again in a sucrose solution which will gradually dissolve it. The

ferric oxide in the saccharate compound is not liable to precipitation by ammonia or alkalis, nor to coloration by tannin, or potassium ferrocyanide, or potassium sulphocyanide. In fact it no longer shows the usual ferric reactions. Ammonium sulphide, on the contrary, gives a precipitate of iron sulphide from such a solution, whilst acids decompose the compound and restore the property of the ferric oxide to give blue colorations with ferrocyanides and red ones with sulphocyanides.

**Lead Saccharate.**

On precipitation of a sucrose solution with an ammoniacal basic lead acetate solution, a precipitate of tribasic lead saccharate is obtained. This may also be prepared by stirring sucrose with water and plumbic oxide; if the proportions are well chosen all the sucrose is deposited as an insoluble tribasic lead saccharate. On suspending this in water, through which a current of carbonic acid is passed, the sucrose can again be liberated.

**Solubility in Sucrose Solution of different Bodies by the Formation of Saccharates.**

Owing to the formation of saccharate a great many substances which are sparingly soluble in water, or even not at all, dissolve easily in sucrose solutions. Lime is for that reason much more soluble in a sucrose solution than in water, and further the calcium saccharate formed thereby possesses the property of dissolving other bodies which do not dissolve in water or in sucrose solutions; among these we may specially mention calcium oxalate, phosphate, and carbonate, and oxides of the heavy metals; when the saccharate is broken up they of course become insoluble again. Sucrose prevents the precipitation of iron, lead, and copper hydroxides, &c., by soda, as those hydroxides immediately pass over into soluble saccharates.

Apart from this formation of saccharates, sucrose solutions dissolve certain other bodies to a greater extent than water; especially towards lime salts of organic acids this property is exhibited in a marked degree, and towards those of sulphuric, sulphurous and carbonic acids to a much less extent, but in case of the simultaneous presence of either alkali or acid their solubility greatly increases, as will be clearly shown in the chapter on *Evaporation*.

**Double Combinations with Salts.**

Sucrose forms double compounds with the salts of alkali metals; those with sodium chloride, sodium iodide, as well as those with potassium chloride, potassium nitrate, and sodium nitrate, are obtained in a crystallized form. Their composition can be represented by the formula

$$C_{22}H_{22}O_{11}NaCl + 2\,H_2O$$

and they can be prepared by allowing a mixture of the sucrose solution with the salt to evaporate slowly at the ordinary temperature in a dessicator over sulphuric acid. These compounds are dissociated in solution, and can be separated into their constituents by dialysis. Besides the few crystallizable double compounds mentioned above, there still exist many others which as yet have not been induced to crystallize and are obtained as viscous syrupy fluids. These combinations, principally those of the organic salts, are of very great importance in

sugar manufacture, and we therefore prefer to discuss them in greater detail in a later chapter on *Molasses*.

**Decomposition of Sucrose by Oxidizing Agents.**

Powerful oxidizing agents readily attack sucrose. Nitric acid oxidizes it with much evolution of gas into saccharic acid, and further into tartric acid, uvic acid, and oxalic acid.

Fehling's solution attacks sucrose only very slowly and after prolonged boiling, that is to say, when it has already become somewhat inverted; pure sucrose is however not attacked. Nor does ammoniacal silver nitrate solution oxidize sucrose solution at the ordinary temperature; on heating, however, silver is deposited as a consequence of the oxidation of the sucrose. Free oxygen does not attack sucrose; in the case, however, of the solution being acid or containing invert sugar it absorbs oxygen with the formation of formic acid. In presence of catalytic substances as spongy platinum, it is oxidized into carbonic acid and water. Finely granulated bone black washed with hot water and treated when still hot with a concentrated sucrose solution at 85—90° C. oxidizes the sucrose in presence of atmospheric oxygen so rapidly, that it becomes caramelized with the liberation of much steam, which reaction is so violent that it sometimes becomes explosive. Ozone, though not attacking neutral sucrose solutions, will oxidize acid or alkaline ones. Hydrogen peroxide causes rapid inversion, especially in presence of iron salts, and after that oxidizes the newly-formed invert sugar.

**Decomposition of Sucrose by Ferments.**

Sucrose is very liable to be broken up by all kinds of ferments. Almost every species of yeast contains a ferment (invertase) that transforms sucrose into invert sugar, which latter afterwards becomes changed by the influence of the fermentative enzymes

**Alcoholic Fermentation.**

of the yeast cells into alcohol, carbonic acid, and certain by-products, thus causing the sucrose as such to totally disappear. Only a very few yeasts do not contain invertase and cannot transform sucrose either directly or indirectly. It has often been suggested to make use of these for removing by fermentation the glucose from syrups or molasses in order to purify them, and thus improve the extraction of the still intact sucrose; but this has not yet been introduced in practice. Some investigators state that during the fermentation of cane juice methyl alcohol is also formed by the action of a special yeast, but detailed researches which the author made on this question* showed that every alcoholic distillate from fermented cane juice, rice, &c., is free from methyl alcohol.

**Lactic and Butyric Acid Fermentation.**

The lactic acid bacteria transform sucrose, especially in presence of lime, into lactic acid, by which process carbonic acid and hydrogen are also formed; this is often followed by the butyric acid fermentation, forming the unpleasant smelling butyric acid from the lactic acid—a phenomenon sometimes met with in practice.

* *Chem. Zeit.*, 1898, No. 9; *Bull. Assoc. Chim. Sucr. et Dist.*, 1898, 115.

Sometimes we find infection by another lower organism, *Leuconostoc mesenterioides*, in cane sugar factories. This rapidly forms a gelatinous body called dextran, as well as much lactic and acetic acids. The dextran is part of the body of the *Leuconostoc* itself, and is principally formed in an alkaline or neutral solution, so that the *Leuconostoc* by its own decomposition products renders the conditions of its development even more troublesome. Heating to the boiling point as well as disinfection with a 1 per cent. ammonium fluoride solution kills the *Leuconostoc*; therefore such a fluid is an excellent remedy against this infection in sugar houses. More particulars about *Leuconostoc* and its removal from manufacture are to be found under the headings *Fructose* and *Clarification*.

**Dextran Fermentation.**

The chief constituent of the gelatinous mass is the dextran, $C_6H_{10}O_5$, which in a pure state appears as a neutral white mass. It does not dissolve in water but swells in it to a thick jelly, which is again precipitated by alcohol. It dissolves in dilute alkalis and then possesses a rotatory power of $\alpha\ D = +230°$. It does not reduce Fehling's solution, and is precipitated from its pseudo-solution in water by basic lead acetate. Acids hydrolyse it under pressure first into unfermentable dextrin and afterwards quantitatively into glucose.

Greig Smith and Steel * describe an organism called by them *Bacillus leraniformans*, which they found in cane juice, in which it formed a gummy substance named by them levan. At the same time succinic and acetic acids were formed. The pure levan dissolves in a small proportion of water to form a mucilage resembling gum arabic, and on the addition of more water the solution becomes white and opalescent A solution of 1 grm. of crude gum in 100 c.c. of water when observed in a 100 mm. tube in a Laurent polarimeter with a monochromatic sodium light gives a reading which is equivalent to a specific rotation of about $\alpha\ D = -40°$. After oxidation with nitric acid of 1·24 specific gravity at 60° C. only oxalic acid is obtained, and neither mucic nor saccharic acid is formed. On treatment with dilute acids in the cold or more quickly on warming, the gum is readily and completely hydrolysed, the sole product being fructose, which is produced in practically the theoretical quantity required by the formula:

**Levan Fermentation.**

$$C_6H_{10}O_5 + H_2O = C_6H_{12}O_6$$

The fungus *Thielaviopsis aethaceticus, Went.* which causes the pine apple disease or black rot of the sugar cane forms, besides alcohol, ethyl acetate from sucrose.

**Ethyl Acetate Fermentation.**

When it is required to ascertain beyond doubt that a solution really contains sucrose, the isolation of this sugar can be effected by means of strontium hydrate by following the rules laid down on page 23. The crystals obtained can be identified by their crystalline form, and by the behaviour of their solution towards polarized light and Fehling's solution before and after hydrolysis by dilute acids.

**Detection of Sucrose.**

* *International Sugar Journal*, 1903, 448.

In the course of time a great many colour tests for sucrose have been suggested, but they all have the disadvantage of not only detecting sucrose but also a number of other kinds of sugar, as they do not apply to the sucrose itself but to its decomposition products, either furfural or humine substances, which, according to the conditions of the reaction, arise from the sugars.

**Colour Tests.**

Among these may be mentioned the α-naphthol reactions. α-naphthol in alcoholic solution when mixed with a trace of sucrose and carefully poured into concentrated sulphuric acid produces a pink-coloured ring where the two liquids meet.

By mixing alcoholic α-naphthol solutions with a trace of sucrose and much concentrated sulphuric acid and heating the mixture on a water-bath, it very quickly assumes a reddish-pink coloration, which, however, likewise appears in the presence of fructose, sorbose, or raffinose. Under similar circumstances β-naphthol yields a reddish-brown and resorcinol a red coloration which, however, is also common to the other already mentioned sugars. As yet there does not exist a characteristic colour reaction for sucrose, so that to actually confirm its presence we are restricted to the characteristic reactions of the substance after its isolation in a pure form.

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## II.—Glucose (Dextrose).

Glucose, besides occurring along with fructose as a product of decomposition due to inversion in over-ripe canes, is a normal constituent of both ripe and unripe cane, and is therefore to be found in all cane juices to a greater or less extent. It is also a very widely distributed constituent of plants in general, and is manufactured commercially in large quantities by the action of sulphuric acid on starch.

**Occurrence.**

Anhydrous glucose, crystallized from its concentrated solution in water, appears in the form of hard columnar crystals, melting at 146°C., while the more common hydrate $C_6H_{12}O_6+H_2O$ occurs in opaque crystal crusts or in big transparent crystals with a melting point of about 86° C. It is impossible to state a fixed figure for its melting point, since at high temperatures the hydrate passes into the anhydride and the varying proportions of these two of course influence the temperature at which it melts.

**Crystalline Form.**

Glucose readily dissolves in water; at 15° C. 100 parts of water dissolve 81·68 parts of the anhydrous compound or 97·85 parts of the hydrate. Glucose is also soluble in alcohol, and the more so in proportion as it is hotter and more dilute. It is very soluble in methyl alcohol, but not in ether nor in acetone.

**Solubility in Water and in other Solvents.**

**Specific Gravity of Glucose Solutions in Water.**

According to Salomon * the specific gravity of glucose solutions in water for the different concentrations is at a temperature of 17·5°C. (where water is 17·5°C=1), as follows:—

| Per cent. | Specific gravity. | Per cent. | Specific gravity. | Per cent. | Specific gravity. |
|---|---|---|---|---|---|
| 1 | 1·00375 | 21 | 1·0800 | 41 | 1·1530 |
| 2 | 1·0075 | 22 | 1·0838 | 42 | 1·1568 |
| 3 | 1·0115 | 23 | 1·0876 | 43 | 1·1605 |
| 4 | 1·0153 | 24 | 1·0910 | 44 | 1·1643 |
| 5 | 1·0192 | 25 | 1·0946 | 45 | 1·1680 |
| 6 | 1·0230 | 26 | 1·0985 | 46 | 1·1716 |
| 7 | 1·0267 | 27 | 1·1020 | 47 | 1·1753 |
| 8 | 1·0305 | 28 | 1·1058 | 48 | 1·1790 |
| 9 | 1·0342 | 29 | 1·1095 | 49 | 1·1825 |
| 10 | 1·0381 | 30 | 1·1130 | 50 | 1·1863 |
| 11 | 1·0420 | 31 | 1·1170 | 51 | 1·1900 |
| 12 | 1·0457 | 32 | 1·1205 | 52 | 1·1935 |
| 13 | 1·0495 | 33 | 1·1240 | 53 | 1·1968 |
| 14 | 1·0533 | 34 | 1·1275 | 54 | 1·2005 |
| 15 | 1·0571 | 35 | 1·1310 | 55 | 1·2040 |
| 16 | 1·0610 | 36 | 1·1348 | 56 | 1·2075 |
| 17 | 1·0649 | 37 | 1·1383 | 57 | 1·2100 |
| 18 | 1·0687 | 38 | 1·1420 | 58 | 1·2148 |
| 19 | 1·0725 | 39 | 1·1456 | 59 | 1·2183 |
| 20 | 1·0762 | 40 | 1·1494 | 60 | 1·2218 |

**Rotatory Power.**

The real value for the specific gravity is not obtained at once when the anhydrous glucose is dissolved in water but one which is a little lower; it increases only after some time, probably owing to the slow transformation into the hydrate. Solutions of the hydrate, however, when freshly made show immediately the same specific gravity as that given by solutions which have stood for some time. Like sucrose, glucose deflects the plane of polarization to the right, though to a lesser degree. At high concentrations the specific rotatory power is somewhat greater than at low ones, but at the concentration in use in sugar-house practice it is pretty well constant and may be stated as $\alpha D = + 52{\cdot}85$ for the anhydrous glucose in a concentration of 14 grms. in 100 c.c.

Taking the rotation of sucrose as 100, then that of the anhydrous glucose is 79·7, and that of the hydrate 72·5. As glucose can be determined by the

* *Berichte*, 121, 2711.

**Influence of Concentration and Temperature on the Rotatory Power.**

polarimeter in the same way as sucrose, and as every degree Ventzke equals 1 per cent. sucrose when 26·048 grms. of the saccharine matter under observation is dissolved in 100 c.c. at 17·5° C. (26 gr. in 100 true c.c. at 4° C.) and read in a 200 mm. tube, so under the same circumstances every degree Ventzke is equal to 1·25 per cent. of anhydrous glucose. Variation in temperature does not influence this rotatory power within those limits which are not exceeded in ordinary laboratory practice.

**Influence of Alkalis and Salts.**

Alkalis, and salts with an alkaline reaction, however, influence the rotation because these bodies either attack glucose or combine with it. Neutral salts when present in considerable quantities reduce it, but in ordinary work no salt concentrations occur to such a degree as to exert any appreciable influence, and they can therefore safely be neglected. Neither acids nor sucrose affect the specific rotation of glucose.

**Birotation.**

The above figures do not apply in the case of freshly made, cold, glucose solutions, as in this case the glucose has a much greater rotatory power, which may even become twice the value just mentioned. After standing for some time at the ordinary temperature, or sooner on being heated, the birotation disappears, and the rotatory power reverts to the constant figure of 52·85°.

Two glucose solutions, containing respectively 1·8194 grms. and 1·1051 grms. of glucose in 20 c.c. of water showed subsequently the following values for the rotation:— *

| Time after being dissolved. | Specific rotatory power observed. | Time after being dissolved. | Specific rotatory power observed. |
|---|---|---|---|
| 5½ minutes | + 105·16° | 7 minutes | + 104·26° |
| 10 ,, | + 101·55° | 15 ,, | + 98·63° |
| 15 ,, | + 96·99° | 30 ,, | + 88·61° |
| 25 ,, | + 87·86° | 60 ,, | + 73·58° |
| 50 ,, | + 72·26° | 420 ,, | + 52·60° |
| 70 ,, | + 63·33° | | |
| 90 ,, | + 59·71° | | |
| 360 ,, | + 52·94° | | |

Glucose, when dissolved in water containing ammonia, shows a constant rotation immediately without any sign of birotation. A concentration of 0·1 per cent. of ammonia is quite sufficient for this purpose, nor is it advisable to use a greater one as strong ammonia will attack the glucose and affect its rotation.

* Parcus and Tollens, *Zeitsch. Rübenzuckerind.*, 40, 841.

The refraction of light caused by glucose solutions is shown in Stolle's table* on refraction given here. The figures refer to observations, of which one series is made 10 minutes and the other 24 hours after the preparation of the solutions. It is seen from these figures that the refractive indices of glucose solutions differ only very slightly from those of sucrose solutions; this has since been confirmed by a large number of other investigators.

**Refraction of Light.**

| Concentration of the Glucose. | | Specific gravity $\frac{17{\cdot}5}{4}$ | | Refractive Index. | |
|---|---|---|---|---|---|
| 0·9967 | 0·9967 | 1·00215 | 1·00215 | 1·33465 | 1·33473 |
| 2·0013 | 2·0013 | 1·00624 | 1·00624 | 1·33588 | 1·33605 |
| 4·0021 | 4·0022 | 1·01383 | 1·01383 | 1·33873 | 1·33890 |
| 8·0058 | 8·0059 | 1·02879 | 1·02881 | 1·34448 | 1·34456 |
| 12·0252 | 12·0257 | 1·04413 | 1·04417 | 1·35017 | 1·35026 |
| 15·9976 | 15·9990 | 1·05882 | 1·05891 | 1·35572 | 1·35581 |
| 19·9896 | 19·9915 | 1·07384 | 1·07394 | 1·36128 | 1·36164 |
| 25·0168 | 25·0193 | 1·09259 | 1·09270 | 1·36825 | 1·36863 |

Glucose when heated above 100° C. assumes a brown coloration; at 170° C. water passes off, and the coloured residue contains, amongst other products, dextrosan or glucosan $C_6H_{10}O_5$, while at a still higher temperature the substance swells, blackens, and gives off a large quantity of gaseous products, leaving a carbonaceous mass, containing caramel, which, when further heated in contact with the air, undergoes total combustion.

**Calorific Value.** The calorific value of glucose as determined in the bomb calorimeter amounts to 3742·6 kg./kg. calories or 6736·7 B.T.U.

**Action of Acids.** Dilute acids exercise no influence on glucose, but more concentrated strong acids, *e.g.*, a 5 per cent. sulphuric acid solution, break it up just like sucrose, causing formation of black humid compounds, a little levulinic and formic acid and a little carbonic acid and carbon monoxide.

**Action of Alkalis.** Dilute alkalis convert glucose, especially at a high temperature, into a mixture of glucose, fructose, and mannose. This process goes on gradually until the glucose and the fructose are represented in approximately equal parts, while the quantity of mannose is very small.

**Converting Power.** Even very weak concentrations of alkalis or alkaline earths have this effect. A quantity of 5 grms. of lime when added to 100 grms. of glucose dissolved in 400 grms. of water is able at a temperature of 70° C. to convert the dextrose into the above-mentioned mixture within a few hours. On dissolving 20 grms. of anhydrous glucose and 10 c.c. of N/1 potassium hydroxide solution in water to a volume of 500 c.c., and heating the mixture to 60° C. the rotation decreases after 10,

* *Zeitsch. Rübenzuckerind.*, 51, 335.

20, 30, 40, 50, 85, and 135 minutes to + 5° 30′, 4° 20′, 3° 10′, 2° 20′, 1° 50′, 0° 43′, and ± 0° 10′ respectively. The specific rotation thus decreases from ± 46 to about 0, while at this temperature comparatively little glucose is decomposed by the action of the dilute alkali. Basic lead acetate, being an alkaline substance, also possesses this power, and so do, at a high temperature, the potassium and sodium salts of organic acids, but here the small amount of dissociated base contained in solution is able to effect the transformation without destroying the glucose.

**Formation of Glucosates.**

Apart from this reaction, alkalis and alkaline earths combine with glucose and form glucosates, compounds of a character analogous to the saccharates already mentioned. Potassium and sodium glucosates are formed by the precipitation of alcoholic glucose solutions with alcoholic potash or soda solutions; their composition is expressed by the formula $C_6H_{11}O_6K$.; they are not crystallizable and are unstable both in the dry state and in solution. Calcium glucosate can be obtained by the precipitation of a glucose- and lime-containing solution with alcohol; this compound is also unstable and is broken up at 50° C. with the formation of colour when exposed to light. Neither neutral nor basic lead acetate throws down glucose from its solution in water, but it is entirely precipitated by ammoniacal lead acetate (*i.e.*, basic lead acetate to which ammonia is added till a precipitate is just about to form).

**Destructive Action of Alkali.**

These glucosates and those of the heavy metals are without special interest to the sugar manufacturer; but a third action of alkali on glucose is the destructive one, which takes place to some extent in the first-mentioned action in which the glucose is transformed. Together with this a part of the glucose is decomposed and if the quantity of alkali is sufficient and the action is continued long enough, all of the glucose is gradually destroyed and transformed chiefly into organic acids.

**Decomposition Products.**

The products of the decomposition formed by this reaction differ according to the concentration, the temperature, and the nature of the alkali. On heating a glucose solution under 70° C. with potash or soda the alkaline reaction disappears, the liquid assumes a brown coloration, and it is found to contain the salts of two acids, which Peligot called glucinic and saccharumic acid. The former of these two is very unstable and decomposes spontaneously, immediately at high temperature, and gradually when cold, into humic, acetic, formic, and carbonic acids, while also some apoglucinic acid is formed. The saccharumic acid is a more stable one and forms readily crystallizable salts; when uncombined it however soon becomes coloured and decomposes into a number of products the nature of which is as yet uninvestigated. This spontaneous decomposition of the acids formed by action of alkaline bodies on glucose accounts for the fact that cane syrups and molasses which at the outset are neutral subsequently always become acid.

At very high temperatures, at boiling point for instance, potash attacks glucose solutions with the formation of alcohol, acetone, acetic and formic acids, and dark coloured humic substances.

At the ordinary temperature alkalis convert glucose without any considerable coloration, by simple molecular disengagement, into much lactic acid, a very stable body which is not liable to spontaneous decomposition. At temperatures under 55° C. lime also forms much lactic acid from glucose, but at higher temperatures the already mentioned acids are formed, which are apt to undergo spontaneous decomposition and become acid. This is the reason why during the carbonatation process of cane juice, where much lime is brought into contact with glucose, the temperature of the limed juice must be maintained under 55° C. so long as the excess of lime is not saturated by carbonic acid.

Baryta forms products analogous to those obtained with lime, but as some of the primary decomposition products possess insoluble barium salts they are thrown down and thus escape further decomposition, so that when working with baryta there is less chance of acid liquids than with lime.

**Double Compounds.** Glucose forms double compounds with salts of a similar character as sucrose. Those with potassium and sodium chloride and with sodium bromide crystallize, but there are still many others which have not yet been obtained in the crystalline form.

Glucose is very liable to oxidation by various reagents. Nitric acid converts it into saccharic acid while at the same time oxalic and tartaric acids are formed.

**Action of Oxidizing Agents.** Free oxygen oxidizes an alkaline glucose solution; ozone leaves glucose intact when in neutral solution, but in an alkaline liquid oxidizes it completely into formic and carbonic acid and water. Hydrogen peroxide causes, especially in presence of a trace of an iron salt, a violent reaction, and which takes place with development of heat, formic, acetic, and tartaric acids and some other compounds being formed.

Glucose, especially in alkaline solution, readily absorbs oxygen, both free and combined, hence the salts of many metallic oxides, such as gold chloride, silver

**Reduction of Cupric Oxide.** nitrate, as well as platinum chloride, mercuric chloride, and bismuth nitrate become reduced by an alkaline glucose solution. Cupric oxide is reduced to cuprous oxide, and this in proportion to the quantity of glucose, provided the conditions are always similar. This property is made use of in the determination of glucose with Fehling's solution, an alkaline solution of cupric hydroxide. In this process the content of a glucose solution is ascertained by the quantity which is required to transform all the cupric oxide contained in a known quantity of the standard copper solution into cuprous oxide, or by weighing the quantity of cuprous oxide which has been precipitated on boiling a known quantity of the glucose solution with an excess of Fehling's solution. During the oxidation the glucose is converted into organic acids, the character of which is not yet fully known.

Iodine dissolved in borax solution slowly oxidizes glucose into gluconic acid, while this reagent does not attack ketoses. Romyn has based a method of determining glucose in the presence of fructose on this reaction.*

**Glucosides.** On introducing gaseous hydrochloric acid into a solution of glucose in methyl alcohol, methyl glucoside is formed and water split off.

$$C_6H_{12}O_6 + CH_3OH = C_6H_{11}O_5O.CH_3 + H_2O$$

This glucoside can more easily be prepared by boiling 1 part of glucose for half-an-hour to an hour in a reflux condenser with 4 parts of acetone-free methyl alcohol which has previously been dehydrated by redistillation over quicklime and then heated with 0·25 per cent. of gaseous dry hydrochloric acid. After that the liquid is heated in a sealed tube in a water-bath for 50 hours, evaporated to one third of its volume, and allowed to crystallize. Besides methyl glucoside also ethyl, propyl, isopropyl, amyl, allyl and other glucosides have been prepared. All these share with the natural glucosides extracted from plants the properties of not reducing Fehling's solution, of being stable towards alkalis, and of becoming hydrolysed by acids with the liberation of the glucose. Numerous attempts to form sucrose by these or similar reactions from glucose and fructose have been unsuccessful.

**Action of Yeast.** Glucose may be decomposed by yeast, and since it needs no previous inversion the fermentation proceeds more rapidly than with the sucrose. The organisms of the lactic and butyric acid fermentations as well as *Leuconostoc* attack it in the same way as with sucrose.

**Detection of Glucose.** When it is necessary to state without doubt that a sugar is actually glucose, we can make use of its property of oxidizing into sucric acid, which is only formed from glucose or glucose containing groups, *e.g.*, sucrose. To this end 5 grms. of the sugar is evaporated on the water-bath with 30 c.c. of nitric acid of 1·15 spec. gravity to a syrupy consistency and the excess of acid expelled by adding water and evaporating again. The residue is dissolved in 20 c.c. of water, carefully neutralized with potassium carbonate while still hot, and a few drops of acetic acid added; this addition is repeated after again evaporating. If glucose has originally been present, the potassium salt of saccharic acid crystallizes, and is drained off on a porcelain plate, re-crystallized from as little water as possible, re-dissolved, neutralized with ammonia, and precipitated by silver nitrate; 5 grms. of glucose yield 2 grms. of the silver salt dried in a dessicator over sulphuric acid; it must, however, not be forgotten that the simultaneous presence of other readily oxidizable sugars can cause glucose to pass over into oxalic acid along with them.

Another characteristic test for glucose is its osazone, which is prepared in the following way:—

**Osazone.** 1 grm. of anhydrous glucose if dissolved in 100 c.c. of water and mixed with 5 c.c. of a solution of 40 grms. of phenylhydrazine and 40 grms. of

* *Zeitsch. Anal. Chem.*, 1897, 349.

glacial acetic acid, and the volume made up to 100 c.c., will yield, after being heated for one hour at 100° C., a crystalline precipitate of glucosazone, the weight of which is, after cooling, filtering, washing and drying, 0·32 grms. Its melting point is 205°, provided that care is taken in the determination to heat the substance rapidly, for if carried out otherwise too high a value is obtained.

**Colour Reactions.** A number of colour tests are yielded by glucose, such as a red coloration with a freshly prepared sulphuric acid solution of α-naphthol, but none of these is characteristic of glucose alone, since such colour reactions, as has already been pointed out when dealing with sucrose, are common to other sugars also.

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## III.—Fructose (Levulose).

In the sap of perfectly ripe sugar cane little or no fructose seems to occur, but it may be found in unripe or overripe canes. In unripe cane it is principally **Occurrence.** present in the parts still growing, from which it disappears as the cane ripens. In dead canes, where the sucrose has already undergone partial inversion, fructose occurs, of course, as one of the products of this inversion.

**Crystalline forms.** Fructose appears in the form of fine hygroscopic needles or of compact crusts of transparent prisms, which are not hygroscopic, and possess a specific gravity of 1·669 at 17·5° C.

**Specific Gravity of Fructose Solutions.** Fructose easily dissolves in water; its solutions possess the following specific gravity values at 17·5° C.

| Per cent. Fructose. | Specific gravity. | Per cent. Fructose. | Specific gravity. |
|---|---|---|---|
| 6 | 1·02150 | 16 | 1·06503 |
| 7 | 1·02575 | 17 | 1·06950 |
| 8 | 1·03012 | 18 | 1·07380 |
| 9 | 1·03447 | 19 | 1·07825 |
| 10 | 1·03870 | 20 | 1·08253 |
| 11 | 1·04303 | 21 | 1·08700 |
| 12 | 1·04747 | 22 | 1·09137 |
| 13 | 1·05175 | 23 | 1·09588 |
| 14 | 1·05620 | 24 | 1·10030 |
| 15 | 1·06053 | 25 | 1·10488 |

It is slightly soluble in cold absolute alcohol, but very soluble in hot and in dilute alcohol, and also in a mixture of alcohol and ether. It is also soluble in methyl alcohol, in glycerin, and in acetone.

**Rotatory Power.** It deflects the plane of polarization to the left, and its levo-rotatory power exceeds the dextro-rotatory power of glucose and sucrose; the rotation is, however, much influenced by temperature, and diminishes when the latter rises.

**Influence of Concentration and Temperature on the Rotation.** In concentrations up to 20 per cent. the specific rotation of fructose diminishes by 0·67° for every degree C. rise in temperature. For a concentration of 10 per cent. the specific rotation is according to Jungfleisch and Grimbert at 20° C. – 90·18° arc degrees or – 134·1 degrees Ventzke. At higher concentrations these values become greater, so the specific rotation at the same temperature for a concentration equal to 5 per cent. is – 95·22 arc degrees or – 143·3 degrees Ventzke.

**Birotation.** Fructose shows birotation but not to such an extent as glucose. Jungfleisch and Grimbert have shown that at 7° C. and for a concentration of 9·75 per cent. a rotation of $\alpha D = -97{\cdot}33°, -96{\cdot}11°, -95{\cdot}11°$, and $-94{\cdot}77°$, is given 35, 55, 75, and 105 minutes respectively after the moment of the preparation of the solution, and that after that the rotation remained unchanged.

**Influence of Foreign Bodies on the Rotatory Power.** The rotatory power of fructose is considerably influenced by all kinds of foreign bodies; it is for instance reduced by alcohol and by hydrochloric acid. Alkalis diminish it too, but this can be ascribed to the formation of fructosates, which causes the amount of uncombined fructose to decrease.

**Optical Refraction.** The values for the refractive indices of fructose solutions are given in the following table by Stolle. The figures in the first column apply to solutions which were obtained 10 minutes after the preparation of the solution, those in the second after six hours, and those of the third after 24 hours.

| Concentration of the Anhydrous Fructose. | | | Specific Gravity $\frac{17{\cdot}5}{4}$ | | | Refractive Index. | | |
|---|---|---|---|---|---|---|---|---|
| 1·0090 | 1·0091 | 1·0091 | 1·00236 | 1·00245 | 1·00244 | 1·33448 | 1·33448 | 1·33448 |
| 2·0100 | 2·0100 | 2·0105 | 1·00610 | 1·00610 | 1·00635 | 1·33596 | 1·33558 | 1·33597 |
| 4·0110 | 4·0016 | 4·0124 | 1·01406 | 1·01420 | 1·01441 | 1·33872 | 1·33854 | 1·33882 |
| 8·0073 | 8·0074 | 8·0091 | 1·02932 | 1·02933 | 1·02955 | 1·34447 | 1·34421 | 1·34456 |
| 12·0250 | 12·0263 | 12·0275 | 1·04513 | 1·04524 | 1·04534 | 1·35008 | 1·34991 | 1·35036 |
| 15·9990 | 15·9999 | 16·0055 | 1·06112 | 1·06017 | 1·06050 | 1·35572 | 1·35545 | 0·35591 |
| 25·0160 | 25·0190 | 25·0187 | 1·09466 | 1·09479 | 1·09478 | 1·36825 | 1·36846 | 1·36814 |

**Behaviour of Fructose on Heating.** On heating dry fructose for some time above its melting point, it becomes partially decomposed and forms an amorphous yellowish mass. On heating it *in vacuo* at 140° or 160° C., water splits off and fructosan (levulosan) remains to the amount of about 90 per cent. of the weight of the fructose used.

Moist fructose is decomposed even at lower temperatures and then gives off water and carbonic acid. It is therefore very necessary when drying substances containing fructose on determining moisture in all cane sugar house products, to carefully avoid decomposing the fructose, otherwise too high a value for the moisture would result. It is advisable to dry such substances first at 60° C. and to afterwards drive off the last traces of water at 100° C. On further heating, fructose gives off much vapour and leaves behind a carbonaceous mass, which on continued heating in presence of the oxygen of the air undergoes total combustion.

**Calorific Value.** The calorific value of fructose as determined in the bomb calorimeter is 3755·0 kg./kg. calories, or 6759·0 B.T.U.

**Decomposition of Fructose on Boiling Aqueous Solutions.** On boiling fructrose with water it is very rapidly decomposed; 4 grms. of fructose boiled in a platinum vessel attached to a reflux condenser for 60 hours with 50 c.c. of water, yield a brown-coloured liquid, having an acid reaction. The rotatory and the reducing powers are thereby diminished by 10 per cent. and about 1 per cent. of the sugar becomes unfermentable.* If the fructose solution is heated to temperatures above 100° C., the decomposition is much quicker and more intense and gives rise to the formation of a number of acids.

**Action of Acids.** Very dilute acids, heated for a long time with fructose solution, change its rotatory power, whilst more concentrated mineral acids (*e.g.*, 5 per cent. hydrochloric or sulphuric acid), split it up into formic, levulinic, and humic acids with the simultaneous formation of furfural and carbonic acid.

Dilute alkalis, as well as potassium and sodium salts of organic acids, exert an analogous action on fructose to that on glucose, and the final products are identical. Especially at high temperatures, fructose is on partial decomposition converted into fructose, glucose, and mannose, the composition of which mixture is the same as that obtained in the transformation of glucose. The strong levo-rotation of the fructose hereby gradually diminishes till the mixture shows no further rotation. During this transformation more acids are formed by decomposition than in the case of glucose, while decomposition is always more rapid with fructose than with the former sugar. In practical work, however, much difference cannot be observed in the products of decomposition of glucose and fructose as both their character and their mutual proportion are very much alike.

**Action of Alkalis.**

* Lobry de Bruijn and Alberta v. Ekestein *Rec. Trav. Chim. Pays Bas* 16.282.

**Alkali Fructosates.** The fructosates of potassium and sodium can be prepared in exactly the same way as the corresponding glucosates; they are extremely hygroscopic and soon become decomposed with the formation of colour. Their general formula is:

$$C_6H_{11}O_6Na.$$

Calcium fructosate is chiefly employed for isolating fructose from its solutions. To this end the solution is cooled down to a few degrees below freezing point and **Calcium Fructosate.** mixed with 6 parts of fresh and very finely powdered quicklime to every 5 parts of fructose present. After that the mixture is stirred and allowed to crystallize. After 24 hours' standing the calcium fructosate is found to crystallize in the form of fine needles, which are centrifugalled, suspended in ice water, and decomposed with carbonic acid, which liberates the fructose. All these operations must be performed in the cold as the fructosate is easily decomposed by heat.

Neither neutral nor basic lead acetate throws down fructose from its solution in pure water, but the last mentioned reagent precipitates this sugar in part so **Lead Fructosate.** long as salts occur in the same solution, with which basic lead acetate forms insoluble compounds. When in this case a precipitate of a lead salt is formed, it carries down a part of the fructose as a plumbic salt. As in the clarification of cane sugar products the addition of basic lead acetate is only to form an insoluble precipitate with compounds in the solutions, this reagent will also eliminate a part of the fructose from the product. For this reason clarification with lead subacetate or basic lead acetate is not applicable when it is necessary to determine the content of reducing sugars in the filtrate. Such solutions are better clarified with neutral lead acetate.

Fructose is quantitatively precipitated by ammonical lead subacetate (*i.e.*, basic acetate of lead to which ammonia is added till a precipitate is just about to form). The precipitate is white at first but becomes pink after some days owing to partial decomposition.

As in the case of sucrose and glucose, fructose combines with salts to form double compounds; of these the following have already been obtained in the crystallized **Double Compounds.** form by evaporating their concentrated aqueous solutions over sulphuric acid: calcium chloride, bromide, and iodide; strontium chloride, bromide, and iodide; and barium iodide. Their formulae are respectively:—

$C_6H_{12}O_6 . Ca Cl_2 + 2 H_2O$; $C_6H_{12}O_6 . Ca Br_2 + 4 H_2O$; $C_6H_{12}O_6 . CaI_2 + 2 H_2O$; $C_6H_{12}O_6 . Sr Cl_2 + 3 H_2O$; $C_6H_{12}O_6 . Sr Br_2 + 3 H_2O$; $C_6H_{12}O_6 . Sr I_2 + 4 H_2O$; and $C_6H_{12}O_6 . Ba I_2 + 2 H_2O$.

These double compounds readily dissolve in water but they are dissociated in aqueous solution, so that their solutions exert a rotation equal to that of the fructose which they contain. Compounds with other salts have not yet been

obtained in the crystalline form, but they undoubtedly exist as will be shown in the chapter on *Molasses*.

Nitric acid oxidizes fructose into formic, oxalic, uvic acid, &c., without the formation of sucric acid. Hydrogen peroxide yields, especially in presence of a trace of an iron compound, acetic, formic and tartaric acid. The oxidation products of fructose by the action of metallic oxides are identical with those of glucose, the only difference in the decomposition being that fructose is more quickly oxidized than fructose. Iodine dissolved in borax solution does not oxidize fructose, and by means of this reaction it may be differentiated from glucose.

**Action of Oxidizing Agents.**

A substance analogous to methyl glucoside is obtained from fructose by dissolving one part of crystallized fructose in nine parts of anhydrous methyl alcohol and adding so much solution of gaseous hydrochloric acid in methyl alcohol that the mixture contains about 0·5 per cent. of hydrochloric acid. The mixture is allowed to stand for 48 hours at 35° C., it is then neutralized with silver carbonate and evaporated. The fructoside does not crystallize nor does it reduce Fehling's solution, but it becomes hydrolysed by boiling with dilute acid with the re-formation of fructose.

**Fructosides.**

Fructose is liable to the action of all the fermentative agents which attack glucose; it ferments with wine or beer yeast, though slower than glucose, but finally yields the same products in the same proportion; it is also attacked by the lactic and butyric acid ferments.

**Action of Yeast.**

The action of *Leuconostoc mesenterioides* is somewhat different for the three sugars under review. *Leuconostoc* forms much more dextran from sterilized sucrose solutions mixed with nutrient salts than from the corresponding glucose and fructose solutions; besides the action commences much more readily in the case of the first-mentioned sugar.

**Leuconostoc Fermentation.**

The rapidity of fermentation of the three sugars will be seen from the tables given here.*

**Rapidity of the Leuconostoc Fermentation.**

| Time. | Glucose. | | Fructose. | | Sucrose. | | |
|---|---|---|---|---|---|---|---|
| | Polarization. | Reducing Sugars. | Polarization. | Reducing Sugars. | Polarization. | Reducing Sugars. Before Inversion. | Reducing Sugars. After Inversion. |
| Immediately.. .. | 31·6 | 7·3 | — 45·0 | 8·8 | 38·7 | 0 | 9·63 |
| After 24 hours .. | 29·5 | 7·1 | — 40·5 | 7·9 | 4·0 | 3·0 | — |
| ,, 2 × 24 hours | 6·1 | 1·5 | 0 | 0·72 | — 2·0 | 0·80 | 1·54 |
| ,, 3 × 24 ,, | 0 | 0·10 | — | — | 0 | 0·25 | 0·25 |

* *Archief voor de Java Suikerindustrie*, 1898, 919.

In continuation of the above observations, a sucrose solution and also a mixture of glucose and fructose solutions were mixed with nutrient salts and calcium carbonate, and sterilized. After neutralizing by adding a few drops of sodium carbonate, the solutions were inoculated with *Leuconostoc* and put aside at 28° C.

Portions of the liquid were analysed daily with the following results:—

| Time. | Mixture of Glucose and Fructose. | | Saccharose. | | |
|---|---|---|---|---|---|
| | Polarization. | Reducing sugars. | Polarization. | Reducing sugars. | |
| | | | | Before inversion. | After inversion. |
| Immediately .. .. | — 2·0 | 8·33 | 37·3 | 0·0 | 9·52 |
| After 24 hours .... | — 2·4 | 2·04 | 4·0 | 2·22 | 4·0 |
| ,, 2 × 24 hours. | + 2·2 | 0·77 | 0·0 | 0·9 | 0·9 |
| ,, 3 × 24 ,, | + 2·2 | 0·77 | 0·0 | 0·3 | 0·3 |

**Varying Behaviour of the three Sugars towards the Dextran Ferment.**

It is clear that in both cases the sugars are fermented in practically the same proportion, but in the case of sucrose much more dextran was formed than with the mixture of the two reducing sugars.

This was confirmed by a direct experiment in which weighed portions of dextran were placed in solutions of sucrose, glucose, and fructose, both with and without addition of sodium and calcium carbonate. After 2 × 24 hours the lump of dextran was weighed again, and the dextran dissolved in the liquid ascertained by weight after precipitation with alcohol.

| Character of the solution. | Initial weight of moist dextran, in grms. | Moist dextran at the end of the experiment, in grms. | Growth of the piece of dextran, in grms. | Dextran dissolved, dried at 100° C, in grms. |
|---|---|---|---|---|
| Sucrose, acid.. .. .. | 8·3 | 10·5 | 2·2 | 5·25 |
| ,, neutralized.... | 10·3 | 22·5 | 12·5 | 13·75 |
| Glucose, acid.. .. .. | 7·5 | 7·8 | 0·3 | 2·90 |
| ,, neutralized.... | 8·5 | 9·4 | 0·9 | 4·10 |
| Fructose, acid .. .. | 6·9 | 6·9 | 0·0 | 2·12 |
| ,, neutralized.... | 7·4 | 7·8 | 0·4 | 2·70 |

This experiment confirms the statement that although *Leuconostoc* can use every one of the three sugars for its growth, yet it is most active towards sucrose, and that a neutral reaction is favourable to the formation of dextran.

**Detection of Fructose.**

The strong levo-rotatory power and the property of forming an insoluble compound with lime are characteristic of fructose, as well as the reaction of yielding neither mucic nor sucric acid on oxidation with nitric acid. But besides these the quantity of osazone which it yields with phenylhydrazine is a good confirmatory test. On heating 1 grm. of anhydrous fructose dissolved in 100 c.c. of water for an hour with 5 c.c. of the phenylhydrazine and glacial acetic acid mixture mentioned on page 33, the same glucosazone, as in the case of glucose, is precipitated, but its quantity is 0·70 grms., or almost twice as much as in the former case. The melting point of the glucosazone is of course also 205° C.

**Methylphenyl Glucosazone.**

Another good characteristic is the methylphenylosazone, for this compound does not form from aldoses at all, and from the ketoses only of fructose in a crystallized form. In order to obtain the crystallized precipitate a solution of 1·8 grms. of fructose in 10 c.c. of water is mixed with 4 grms. of methylphenylhydrazine and so much alcohol that a limpid solution is obtained. Now 4 c.c. of 50 per cent. acetic acid are added, the mixture is heated from 5 to (at a maximum) 10 minutes in a water bath and allowed to cool in a beaker covered with a glass plate. After some time, not exceeding two hours, reddish crystals amounting to 80 per cent. of the fructose used are formed; these may be recrystallized from hot 10 per cent. alcohol and should have a melting point of 150° C.

**Colour Reactions.**

Fructose, even in very dilute solution, may be detected with great ease by a great number of colour reactions, but here also most of these tests do not apply solely to fructose but likewise to other sugars, for instance sucrose, which in most of the reactions is hydrolysed first into glucose and fructose, which latter then give the reaction as though originally present. Thus the colour reactions with the $\alpha$- and $\beta$-naphthol and with resorcinol and sulphuric acid apply as well to fructose as to raffinose, sucrose, and the other ketose, sorbose.

The colour reaction with ammonium molybdate and acetic acid is the only one which can be claimed to be characteristic for fructose. It should be noted that, except acetic acid, no other organic or inorganic free acid must be present in the solution. On adding to 10 c.c. of a 4 per cent. ammonium molybdate solution, 10 c.c. water, 0·2 grms. of glacial acetic acid and a trace of fructose, and heating the mixture in a water bath at 95—98° C., after three minutes a deep blue coloration appears, owing to the reduction of the molybdic acid. Other sugars produce this coloration less rapidly and less intensely.

This reaction does not occur in the presence of free acids other than acetic acid; these may be removed by adding lead acetate, precipitating the excess of lead with sodium carbonate, and acidifying the filtrate with acetic acid.

# IV.—Invert Sugar.

Although invert sugar is not a simple sugar but consists of a mixture of equal parts of glucose and fructose, it occurs so largely that some particulars of it may be mentioned under this separate heading.

**Preparation.** Invert sugar is prepared commercially by heating a sucrose solution with a very small quantity of acid until the solution, when examined by the polarimeter, shows the levo-rotation corresponding with its concentration.

Herzfeld* calculated the following table for the levo-rotation of a completely hydrolysed sucrose solution for different concentrations at the temperature of 20° C.

| Per cent. Sucrose. | Per cent. Invert sugar obtained. | Levo-rotation. | | $\alpha$ D 20. |
|---|---|---|---|---|
| | | In ° Ventzke. | In Arc degrees. | |
| 10 | 10·53 | 12·2 | 4·22 | — 20·04 |
| 11 | 11·58 | 13·5 | 4·65 | — 20·08 |
| 12 | 12·64 | 14·7 | 5·08 | — 20·11 |
| 13 | 13·68 | 16·0 | 5·52 | — 20·15 |
| 14 | 14·74 | 17·2 | 5·95 | — 20·19 |
| 15 | 15·79 | 18·5 | 6·39 | — 20·23 |
| 16 | 16·84 | 19·9 | 6·83 | — 20·27 |
| 17 | 17·90 | 21·0 | 7·27 | — 20·30 |
| 18 | 18·95 | 22·4 | 7·71 | — 20·34 |
| 19 | 20·00 | 23·6 | 8·15 | — 20·38 |
| 20 | 21·05 | 24·9 | 8·60 | — 20·42 |
| 21 | 22·10 | 26·2 | 9·04 | — 20·46 |
| 22 | 23·16 | 27·5 | 9·49 | — 20·49 |
| 23 | 24·21 | 28·8 | 9·94 | — 20·53 |
| 24 | 25·26 | 30·1 | 10·39 | — 20·57 |
| 25 | 26·32 | 31·4 | 10·58 | — 20·61 |

An invert sugar solution for analytical purposes is prepared, according to Soxhlet's method, by dissolving 9·5 grms. of sucrose in 700 c.c. of hot water, heating it with 100 c.c. of N/5 solution of hydrochloric acid for half-an-hour on a water-bath at 100° C., cooling rapidly to 20°, neutralizing it with the required amount of N/5 sodium hydroxide solution, and making up with water to 1000 c.c., by which operation a solution of exactly 1 per cent. of invert sugar is obtained.

* *Zeitsch. Rübenzuckerind.*, **37**, 907.

**Rotatory Power.** The specific rotation of invert sugar is equal to the difference in those of the two components and is consequently much affected both by differences in concentration and in temperature.

**Influence of the Temperature on the Specific Rotation.** Lippmann* found these figures for concentrations of 17·12 invert sugar (equal to 16 38 sucrose).

| Degrees C. | 0 | 10 | 20 | 30 | 40 | 50 | 60 | 70 | 80 |
|---|---|---|---|---|---|---|---|---|---|
| $\alpha D$ | — 27·9 | — 24·5 | — 21·4 | — 18·0 | — 15 2 | — 12·0 | — 8·5 | — 5·8 | — 2·0 |

which agree very well with those calculated by Tuchschmid's formula :—

$$\alpha \frac{t}{D} = - (27{\cdot}9 - 0{\cdot}32\ t).$$

According to this formula there exists a temperature, viz., 87° C., at which the rotation of invert sugar is zero ; of this property use is made in sugar analysis, although at so high a temperature invert sugar is liable to be decomposed, and the figures for the rotation thus obtained will therefore not be entirely reliable.

**Influence of Concentration on Rotation.** At higher concentrations the levo-rotation of invert sugar increases as will be shown by Bomtraeger's figures for a temperature of 20° C.

| for conc. = | 5 | 10 | 15 | 20 | 25 | 30 | 40 | 50 | 60 |
|---|---|---|---|---|---|---|---|---|---|
| $\alpha_D^{20}$ = | — 19·75 | — 20·04 | — 20·32 | — 20·58 | — 20·84 | — 21·08 | — 21·53 | — 21·94 | — 22·30 |

In such experiments it should be remembered that by considerably diluting the hydrolysed solution, the specific rotatory power is at first further to the left than after standing some time. In these determinations one must therefore take the precaution to dilute the sucrose solution, previous to the inversion, to about the same concentration as that at which it will be polarized after that operation.

Unlike the polarization of the simple sugars, in which the commonly occurring quantities of salts or acids do not affect the rotatory power to any noticeable extent, that of the invert sugar is usually much influenced by acids or salts, since it is mixed for the purpose of the inversion, with large percentages of hydrochloric acid.

Grubbe† represents the influence of hydrochloric acid on the rotation of invert sugar at 20° C. at a concentration of hydrochloric acid of from 0·3 per cent. (S) by the following formula :—

$$\alpha_D^{20} = - (19{\cdot}55 + 0{\cdot}32621\ S).$$

Herzfeld's prescription‡ requires 5 c.c. of hydrochloric acid of 1·188 sp. gr., corresponding to 6 grms. of 38 per cent. acid or to 2·2 grms. of hydrochloric gas, which is dissolved in a volume of 100 c.c. This concentration will therefore, according to the above formula, increase the rotatory power of the invert sugar by $0{\cdot}32621 \times 2{\cdot}2 = 0{\cdot}72°$.

* *Berichte*, 13, 1823. † *Zeitsch. Rübenzuckerind.*, 1884, 1356.
‡ *Zeitsch. Rubenzuckerind.*, 40, 194, 208, 443.

The influence which is exerted by salts is still more considerable than that of the free acid. Since therefore the presence of hydrochloric acid, the concentration of the solution, and the temperature, are factors which have a considerable influence on the rotation of invert sugar, great care should be taken when polarizing sugar products after inversion, to exactly adhere to the prescribed directions as to weight, concentration, and temperature, and to use the formula which is adapted to these conditions.

The action of acids, bases, &c., on invert sugar is of course the same as has been described for glucose and fructose.

In this work we adopt the system of grouping the glucose, the fructose, and the mixture of both these sugars known as invert sugar, under the common name of "reducing sugars." The amount of these reducing sugars present is determined from the quantity of copper oxide reduced, without making any distinction between the individual sugars in the mixture. Wherever, therefore, in this work the term "reducing sugars" is used, it always means that sugar or mixture of sugars which has the property of reducing Fehling's solution.

**Nomenclature.**

The explanation of the reason why we have adopted the terms glucose, fructose, and reducing sugar, instead of the more widely used expressions dextrose, levulose, and glucose, is given in the Preface.

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## V.—Starch.

Starch occurs in the chlorophyll-bearing cells of the leaves, around the bundles in the leaf-sheaths and around those of the top of the stem, whilst in the adult joints little or no starch is found.

It occurs in very minute granules. It produces a blue colour on treatment with a solution of iodine in potassium iodide.

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## VI.—Fibre.

All the solid substances of the cane are grouped under the name of "cellulose" or "fibre." The first mentioned term is not correct, as the fibre, properly speaking, only partly consists of cellulose, the other constituents being lignin and a pentosan in proportions which vary according to the species of the cane and its age, as will be seen from the table given here.

**Occurrence.**

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| | Black Java. | Seedling No. 36. | Seedling No. 100. | Seedling No. 103. | Seedling No. 105. | Seedling No. 139 | Seedling No. 247. | Black Java. | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | | | 5 | 6 | 9 | 12 |
| | | | | | | | | Age in months. | | | |
| Fibre .. .. .. | 55·94 | 62·57 | 53·68 | 49·29 | 58·39 | 56·76 | 56·22 | 38·36 | 39·75 | 41·36 | 50·3 |
| Pentosan.. .... | 22·23 | 23·68 | 23·32 | 21·46 | 22·54 | 24·08 | 23·80 | 25·70 | 28·50 | 30·82 | 31·50 |
| Ash .. .. .. | 1·98 | 1·47 | 3·49 | 3·12 | 2·23 | 1·68 | 1·98 | 3·45 | 3·35 | 4·02 | 3·95 |
| Nitrogenous Substance, &c. | 2·0 | 2·0 | 2·0 | 2·0 | 2·0 | 2·0 | 2·0 | 2·0 | 2·0 | 2·0 | 2·0 |
| Lignin (by difference) | 17·75 | 10·28 | 17·51 | 24·13 | 14·84 | 15·48 | 16·00 | 30·49 | 26·40 | 21·80 | 12·25 |

By "fibre" is understood that portion of the vegetable tissue which resists boiling with first 1·25 per cent. sulphuric acid for one hour and then with 1·25 per cent. solution of caustic soda for the same period of time.

The pentosans are determined by Tollen's method in which the substance is distilled with hydrochloric acid, the distillate neutralized with sodium carbonate, then acetic acid and a solution of phenylhydrazine acetate added, and the precipitated furfural hydrazone finally weighed.

Cellulose may be hydrolysed by sulphuric acid and is thereby converted into glucose. The pentosan which may be isolated from the fibre by extraction with dilute soda solution and precipitation with acetic acid and alcohol is, when freshly precipitated, soluble in hot water, but on cooling the solution becomes opaque and gelatinous. Alcohol does not immediately precipitate the aqueous solution, but only after the addition of a little acetic acid or soda solution. The aqueous opaque solution becomes clear after addition of soda or potash, and these can dissolve still more of the gum, but it is precipitated by excess of lime or baryta and by lead acetate.

**Properties of Cellulose.**

**Properties of the Pentosan.**

After being dried, the gum is only slightly soluble in water, but it completely dissolves, though slowly, in soda solution.

The solution of 5 grms. of cane gum in 100 c.c. of water obtained by the addition of as little soda as possible is slightly opalescent but can be polarized after being allowed to subside for some days. It deflects the plane of polarization strongly to the left, and to such an extent that the specific rotation of the organic matter, free from moisture and ash, is about $\alpha D = -80°$. On hydrolysis with hydrochloric acid

**Rotatory Power of the Pentosan.**

**Composition of the Pentosan.**

the gum yields (calculated to 100 parts of dry and ash-free material) 90 per cent. of sugar, chiefly xylose, accompanied by a small proportion of another sugar possessing a more positive rotatory power, probably arabinose or glucose. On distillation with hydrochloric acid of 1·06 specific gravity the pentosan yields 46·3 per cent. of furfural which corresponds to 88·3 per cent. of xylan. Oxydation with nitric acid does not produce mucic acid but trioxyglutaric acid, a proof that the cane gum consists of xylan accompanied perhaps by araban. Browne* found 75 per cent. xylan, 24 per cent. araban, and 1 per cent. galactan.

**Combinations of Cane Gum.**

The gum enters into combination with bases. The soda compound is up to now the only one investigated; it dissolves in water to a viscous liquid, but is insoluble in alcohol. The soda content is 10 per cent. which corresponds with the formula $2\,C_5H_8O_4$. NaOH.

In English and American publications we often find the term "gum" used for other bodies which properly speaking do not belong to the class of gummy matters, and might better be tabulated under the name of "organic non-sugar." In the *Hawaiian Planters' Monthly*, 1906, 374, Peck gives the following definition of what he understands as gum:—

"By this term is indicated all the impurities precipitated by subacetate of lead, and includes, naturally, a great many substances other than true gums, such as glucinic, melassinic and succinic acids, and perhaps partially aspartic acid. The precipitate, produced as in the sucrose and glucose determinations, with an exactly sufficient amount of the lead solution, and the addition of a few drops of acetic acid, was thoroughly washed first by decantation and then on the filter, until the washings gave no test for sugars with $\alpha$-naphthol. The precipitate was then suspended in water and decomposed with sulphuretted hydrogen, filtered, concentrated, and weighed in a platinum dish. This was then ignited, and the weight of the ash deducted."

Cane fibre is not insoluble in water since on prolonged ebullition it gradually decreases in weight.

**Solubility of Constituents of the Cane Fibre in Water.**

Shorey found that cane fibre from which all the constituents of the juice had been withdrawn by extraction with cold water yielded a soluble substance to boiling water, and the more so the longer the treatment with hot water was continued. A quantity of finely divided cane, which after extraction with cold water contained 10 per cent. of fibre, only contained 9·3 per cent. of that constituent after boiling with water for five minutes, and no more than 8·8 per cent. when the boiling was continued for two hours. So that during that time no less than 12 per cent. of the fibre were dissolved and carried off. Krüger† boiled 50 grms. of finely shredded cane repeatedly with 200 c.c. of water and dried the residue

* *Sugar Cane*, 1896. † *Mitteilungen der Versuchs Station, Kagok II.*, 4.

after 10, 15, and 20 extractions. The following table shows us that after 20 extractions the fibre content was 0·43 per cent. less than after 10, and that hot water therefore continuously dissolves parts of the cane fibre.

| No. | 10 × *a.* | 15 × *b.* | 20 × *c.* | Difference, *a*—*c.* | 10 × *a.* | 15 × *b.* | 20 × *c.* | Difference, *a*—*c.* |
|---|---|---|---|---|---|---|---|---|
| I. .. .. .. .. | 9·94 | 9·75 | 9·46 | 0·48 | 10·15 | 9·92 | 9·71 | 0·44 |
| II. .... .. .... | 9·98 | 9·78 | 9·59 | 0·39 | 10·18 | 9·93 | 9·74 | 0·44 |
| III. .. .. .. .. | 10·13 | 9·92 | 9·73 | 0·40 | 10·04 | 9·84 | 9·61 | 0·43 |
| Average .. .... | 10·01 | 9·81 | 9·59 | 0·42 | 10·12 | 9·90 | 9·69 | 0·44 |

The writer has stated that the constituent which hot water dissolves from the fibre has a decided dextro-rotatory power. For this reason the sucrose content of bagasse is found to be too high if the boiling is too prolonged. A period of ten minutes, if 30 grms. of bagasse and 300 c.c. of water are used, is quite sufficient to extract all the sugar, and at the same time does not cause any appreciable quantity of the fibre to pass over into the liquid. Owing to the fact that hot water extracts some of the fibre, the determination of the fibre content in sugar-cane and in bagasse by extraction with boiling water is apt to yield lower results for that constituent. We prefer extraction with alcohol of the shredded cane which has previously been washed with cold water in order to remove albuminoids and similar bodies belonging to the juice, which are precipitable by alcohol and are thus capable of causing the weight of the insoluble fibre to be too high.

It is especially in the case of young cane that the fibre content found by water extraction differs much from that determined by extraction with alcohol.

| | Age of the Cane in Months. | | | |
|---|---|---|---|---|
| | 5 | 6 | 9 | 12 |
| Fibre by water extraction .. .. | 7·36 | 7·99 | 8·38 | 10·52 |
| Fibre by alcohol extraction .... | 8·0 | 8·65 | 8 35 | 10·94 |
| Difference on 100 parts of fibre.. | 8·2 | 7·5 | 5·3 | 3·9 |

Water rendered alkaline with lime also dissolves much gummy matter from the fibre of unripe cane, but very little from that of ripe and overripe canes. Caustic soda solutions, on the contrary, dissolve much gummy matter from cane fibre. Particulars regarding the removal of the gum by clarification will be found under the heading *Clarification.*

According to Browne and Blouin the lignin in cane fibre has the same constitution which Lindsey and Tollens ascribe to the lignin from wood, viz., $C_{24}H_{26}(CH_3)_2O_{10}$. The former investigators report the following constitution for cane fibre (protein, ash, fat, &c., excluded): —

| | Per cent. |
|---|---|
| Cellulose (including oxycellulose) .. .. .. .. .. | 55 |
| Xylan .. .. .. .. .. .. .. .. .. .. .. .. .... | 20 |
| Araban .. .. .. .. .. .. .. .. .. .. .. .. .. | 4 |
| Lignin .. .. .. .. .. .. .. .. .. .. .. .... | 15 |
| Acetic acid .. .. .. .. .. .. .. .. .. .. .. | 6 |
| | 100 |

**Colloid Water.** The fibre does not occur in the cane in an anhydrous state, but contains a certain amount of sugar-free colloid water of hydration, so that in the living organism of the cane stalk the fibrous parts are found sugar-free with their own water content along with the sugar-containing parenchyma cells.

We have concluded from a series of experiments that the colloid-water amounts to 20-30 per cent. of the weight of the dried fibre.

**Calorific Value.** As the cane fibre forms the principal source of fuel for the cane sugar industry, the determination of its calorific value is of considerable importance. This value is given here for seven different varieties.

| Fibre of the Varieties. | I. | II. | III. | IV. | V. | VI. | VII. |
|---|---|---|---|---|---|---|---|
| | | | Calorific Value in B.T.U. | | | | |
| | Moisture | Ash. | On the moist fibre. | Calculated to dry fibre from I. & III. | Calculated to dry organic substance from II. & IV. | On dried fibre. | Calculated on dry organic substance from II. & III. |
| Black Java, 1906 .. .. | 9·60 | 2·05 | 7587 | 8388 | 8559 | 8172 | 8370 |
| Seedling No. 247 ..... | 9·12 | 1·85 | 7686 | 8460 | 8620 | 8325 | 8482 |
| ,, ,, 36.. .. | 8·32 | 1·42 | 7695 | 8492 | 8449 | 8171 | 8341 |
| ,, ,, 100..... | 8·46 | 3·03 | 7553 | 8249 | 8256 | 7996 | 8246 |
| ,, ,, 105.. .. | 8·64 | 1·79 | 7616 | 8334 | 8503 | 8125 | 8273 |
| ,, ,, 139..... | 9·26 | 1·42 | 7540 | 8329 | 8442 | 7862 | 7974 |
| Black Java, 1907 .. .. | 8·69 | 1·68 | 7772 | 8514 | 8681 | 8154 | 8293 |

Column V. shows the calorific value for the calculated organic substance, ascertained in the moist samples and calculated on 100 parts of dry and ash-free substance. In column VII. is given the same value but this time determined in the

previously dried samples, and again calculated to 100 parts of dry and ash-free substance. The figures for these latter values are all a little low, which, as all the water driven off was merely hygroscopical water, is difficult to explain, because in that case the figures in columns V. and VII. should have been identical. All of the figures stated are averages of three determinations in which very concordant results were obtained, and because in every one of the cases the difference is on the same side, we may safely conclude to their having one common cause. Whatever this may be is not known yet, but as the fuel is used in the moist and not in the anhydrous condition, the figures of column V. apply more to the conditions met with in practical working. Therefore we suggest using the figures of column V. when it is necessary to have a base for the calculation of heat produced, consumed, and lost in a sugar factory, or for similar calculations, and then we can take the average figure of 4751 kg./kg. calories or 8552 B.T.U.

Coates* found for the calorific value of Louisiana cane 8325 B T.U. or about the same as found in Java.

When making such calculations it must not be forgotten that these figures represent the highest values, as the determinations were made at the common temperature in the calorific bomb, where the latent condensation heat of the water, both already existing and that formed by the combustion, is included in the figure for the calorific value. This heat escapes, however, in practical working, when the vapour is driven off into the chimney and thus carries away that latent heat. In calculations of the real calorific value we therefore subtract from the calculated calorific effect of the sucrose and fibre the figure for the latent heat of all the water, that which already existed in the fuel as well as that which is formed by combustion of the organic substances.

**Elementary Constitution of Cane Fibre.**

The elementary constitution of a few samples of cane fibre calculated for 100 parts of water and ash-free substance is as follows:—

| Fibre of the varieties. | Carbon. | Hydrogen. | Oxygen. | Nitrogen. | Calorific value. | | Ratio H : O. |
|---|---|---|---|---|---|---|---|
| | | | | | V. | VII. | |
| Seedling No. 36.. | 48·45 | 6·45 | 44·94 | 0·16 | 8449 | 8341 | 6·97 |
| ,, ,, 100.. | 47·88 | 6·55 | 45·39 | 0·18 | 8256 | 8246 | 6·93 |
| ,, ,, 105.. | 48·14 | 6·43 | 45·25 | 0·18 | 8503 | 8273 | 7·04 |
| ,, ,, 139.. | 47·13 | 6·30 | 46·43 | 0·14 | 8442 | 7974 | 7·37 |
| Black Java ... | 46·82 | 6·67 | 46·33 | 0·18 | 8681 | 8293 | 6·94 |

* *Louisiana Planter*, 1906, 236.

Cane fibre, therefore, closely resembles wood, as may be seen from the enumeration of the composition of several kinds of wood, to be found in Beilstein's *Organische Chemie* I. 1078.

**Comparisons between Cane Fibre and Wood.**

| Kind of wood. | C. | H. | N. | O. | O : H. | Ash. |
|---|---|---|---|---|---|---|
| Yoke Elm .... | 49·48 | 6·08 | 0·84 | 43·60 | 7·17 | 1·62 |
| Beech .. .. | 49·89 | 6·07 | 0·93 | 43·11 | 7·10 | 1·06 |
| Poplar .. .... | 50·31 | 6·32 | 0·98 | 42·39 | 6·71 | — |
| Birch .. .. .. | 50·61 | 6·23 | 1·12 | 42·04 | 6·75 | 0·85 |
| Oak .. .... | 50·64 | 6·03 | 1·28 | 42·05 | 6·97 | 1·65 |
| Willow .. .. | 51·75 | 6·19 | 0·98 | 41·08 | 6·62 | 2·00 |

The resemblance becomes still more pronounced on comparison of the calorific values of wood and cane fibre. Though not properly belonging to this series, the figures for the calorific value of cane trash (dry leaves) are given here to make our list as complete as possible.

| Kind of Material. | Moisture. | Calorific value in B.T.U. | |
|---|---|---|---|
| | | Undried. | On 100 parts of dry material. |
| Beech .. .. .. .. | 13·0 | 7502 | 8624 |
| Fir .. .. .. .. .... | 12·2 | 7960 | 9067 |
| Oak .. .. .. .. .. | 13·3 | 7182 | 8283 |
| Pine .. .. .. .... | 11·8 | 8073 | 9157 |
| Old Teak .. .. .. .. | 9·58 | 8114 | 8955 |
| Young Teak .. .. .... | 29·41 | 5947 | 8424 |
| Cane Trash (leaves) . | 10·46 | 6663 | 7807 |

**Difference in Comparison of Fibre.**

In chemical composition the fibre of the various cane varieties does not show considerable differences, but its physical aspect does. The fibre from cane having a high fibre content is much more compact than that of cane possessing only a low fibre content. This is shown by the table given here, containing the weight in grms. of 100 c.c. of loosely shaken, finely chopped, air dried cane fibre of various kinds having differing fibre contents.

| Cane variety. | Per cent. fibre in the cane. | Weights in grms. of 100 c.c. fibre. | Number of analyses from which the average is calculated. |
|---|---|---|---|
| Seedling No. 100 .. .. | 9·77 | 5·45 | 3 |
| Black Java .. .. .... | 10·38 | 6·71 | 9 |
| White Manilla .. .. | 11·80 | 6·38 | 3 |
| Seedling No. 247 .... | 13·85 | 6·97 | 8 |
| ,, ,, 139 .. .. | 14·16 | 7·30 | 5 |
| ,, ,, 33A .... | 15·59 | 7·23 | 3 |
| ,, ,, 36 .. .. | 15·72 | 4·95 | 8 |

**Composition of Ash from Cane Fibre.** Apart from the constituents already mentioned, the fibre obtained from cane by extracting all the other substances with alcohol still contains about 1 per cent. of albuminoid matter, a colouring substance which assumes a yellow colour when the fibre is moistened with an alkaline liquid, and from 1 to 3 per cent. of ash, of which the composition is given here:—

| | |
|---|---|
| Silica .. .. .. .. .. .. .. .. .. .. .... | 80·57 |
| Iron and calcium phosphates.. .. .. .. .. .. | 6·87 |
| Calcium carbonate .. .. .. .. .. .. .... | 0·86 |
| Potassium carbonate and hydroxide .. .. .. .. | 11·70 |
| | 100·00 |

## VII.—Pectine.

**Occurrence.** Some pectine is always found in cane juice; in certain varieties it occurs more than in others, though the composition of the soil also seems to influence its occurrence. In the fibrous part of the cane, however, it is only very sparingly, or not at all, represented.

Pectine is only partially removed by clarification, and therefore it may be expected in the clarified juice and in the molasses to a greater or lesser extent.

**Properties.** It is soluble in water but insoluble in acidulated alcohol, it does not yield sugar on being heated with dilute acids, nor furfural on distillation with hydrochloric acid. Its lime salt is somewhat soluble in water and more so in sugar solution. Lead acetate precipitates pectine from its aqueous solution. On evaporating 5 grms. of pectine from cane juice with 60 c.c. of nitric acid of 1·15 sp. gr. about 1 grm. of mucinic acid is obtained, corresponding to a content of about 30 per cent. of galactane groups in the pectine.

# VIII.—Organic Acids.

The organic acids of the sugar cane are glycolic, oxalic, malic, succinic, tannic, and in dead cane (as a product of decomposition) also acetic acid. Some investigators report having also detected citric, tartaric, and aconitic acids, but their allegations have not as yet been confirmed. In ripe canes the quantity of organic acid is but very small, and does not exceed 0·15 per cent., of which quantity 0·06 per cent. is present as free acid, and the rest combined with potash.

**Glycolic Acid.**

Glycolic acid occurs among other constituents in unripe grapes, and is also detected in beet juice bottoms after the juice has been heavily limed and has subsided. Its lime salt is soluble; the acid therefore is not removed by clarification, and it may be traced in the molasses in so far as it has not become decomposed during manufacture.

**Oxalic Acid.**

Oxalic acid is found in a great many plants, among others in beetroot leaves, wood sorrel, &c., whilst it is to be found as calcium oxalate in a number of fruits, tubers, &c. Calcium oxalate is insoluble in water, but dissolves in a sugar solution, especially when this is dilute and alkaline. On concentration it becomes insoluble and is therefore deposited in the last vessels of the evaporation plants in sugar houses as incrustation, while it is no longer found in the syrup.

**Malic Acid.**

Malic acid is found in a great many fruits and juices; it is a very stable acid and does not become decomposed in the course of manufacture: its lime salt is soluble in water, so that the acid is not removed by defecation of cane juice with lime.

**Succinic Acid.**

Succinic acid, like malic acid, is a very stable acid, and like it possesses a soluble lime salt.

**Acetic Acid.**

Acetic acid is only to be found in dead or diseased canes as a product of the decomposition of the sugar. Its lime salt is soluble, and therefore remains in the juices after liming; it has the disadvantageous property of being very hygroscopic, and this is one of the reasons why sugar yielded by damaged or dying canes often becomes moist again after being well dried.

Tannic acid of unknown composition occurs in very minute quantities in young canes and in the green tops of ripe ones. The quantity is somewhat insignificant, and it combines with albumen immediately after the crushing, forming an insoluble precipitate, thus it does not interfere with the further stages of manufacture.

**Acids produced by the Action of Lime on Reducing Sugars.**

The acids mentioned here originate from the cane and pass over into the juice, but during manufacture large quantities of organic acids are produced by the action of lime on the reducing sugars, and on sucrose. Their properties are already mentioned on page 30 under the heading *Glucose*.

## IX.—Cane Wax.

**Occurrence.** On the outside of the rind of the cane, especially near the nodes, we find a more or less considerable layer of wax, which is more conspicuous in some varieties than in others.

**Properties.** This wax melts at 82° C., boils at 146° C., is insoluble in water and in cold alcohol, slightly soluble in cold ether or chloroform, but dissolves easily in hot alcohol, ether, and chloroform, and in benzene.

Properly speaking, it is not a wax but a saturated alcohol, having 24 atoms of carbon :

$$C_{24}H_{50}O.$$

On heating, it is split up into a hydrocarbon and water.

$$C_{24}H_{50}O = C_{24}H_{48} + H_2O.$$

---

## X.—Colouring Matter.

**Occurrence.** In the peripheric strata of the rind of the sugar cane we find cells containing the colouring matter, which causes the differing coloration of **Chlorophyll.** the cane. Besides chlorophyll, which in every case forms the ground colour, the presence of anthocyan has been detected in black and purple **Anthocyan.** canes When the canes are crushed, the colouring matter passes over into the juice and imparts its colour to it; thus the juice from black cane is much darker than that from white and yellow.

**Incrustating Colouring Matter.** Next to this we find in the cane another colouring matter, with which the fibre is impregnated. This is colourless in a neutral state, but becomes yellow on contact with alkaline liquids. It is so firmly connected with the fibre that it is impossible to remove it totally by extraction.

This colouring matter, the composition of which is still unknown, is very sparingly soluble in water, but more so in alcohol; whilst it is insoluble in ether. When exposed to the air, it changes into a red colouring matter, which is frequently met with in diseased or dead canes. On further oxidation it is broken up into a dark brown substance.

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# XI.—Nitrogenous Bodies.

Sugar cane contains very little nitrogenous substance; only the leaves and the green tops contain a rather considerable quantity, but in the ripe parts of the stalk only the buds are provided with it. A perfectly ripe cane, without the leaves, contains an average of 0·05 per cent. nitrogen, and the whole plant, without the roots, up to 0·08 per cent. Juice filtered through cloth was found to contain from 0·018 to 0·062 per cent. of nitrogen, being 0·036 on the average, and of this small amount a considerable portion was not dissolved, but in the form of a colloid, for the amount of nitrogen decreased greatly after filtration through filter paper.

**Occurrence.**

**Composition of the Nitrogenous Substances.**

According to Brown and Blouin* the various nitrogenous bodies of sugar cane may be stated as follows:—

| | Percentage of Cane. |
|---|---|
| Albumen (coagulable and soluble in pepsin) .. .. | 0·059 |
| Nuclein, &c. (coagulable and insoluble in pepsin) .... | 0·040 |
| Albumoses and peptones (not coagulable) .. .. .. | 0·033 |
| Amido acids (aspartic acid) .. .. .. .. .... | 0·145 |
| Amido acid amids (asparagin) .. .. .. .. .. | 0·232 |
| Ammonia .. .. .. .. .. .. .. .. .. .... | 0·008 |
| Nitric acid .. .. .. .. .. .. .. .. .. .. | 0·071 |
| Total nitrogenous bodies .. .. .... | 0·588 |

The author remarks, however, that the above percentages are subject to considerable variations according to the age and variety of the cane, manner of fertilization, and cultivation.

Other investigators report having found glycocol (amido-acetic acid) in the cane, but at any rate its quantity was very small, amounting to about 0·05 per cent. of the weight of the cane.

**Bases.** Finally, small amounts of xanthin bases, chiefly guanin, are isolated from cane juice.

The amount of nitrogenous bodies occurring in exhausted molasses, in which product the impurities from the juice have of course accumulated, so far as they have not been deposited during manufacture, is likewise very small and the total amount of nitrogen seldom exceeds 0·2 per cent. of the weight of the molasses.

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* *Louisiana Exp. Station Bulletin*, No. 91, page 5.

# XII.—Incombustible Matter or Ash.

Sugar cane is a plant that absorbs very little mineral substance from the soil; this is very clearly to be deduced from the minute quantity of ash it contains.

**Occurrence.** The constituents vary in quantity between rather wide limits according to the quality of the soil, the manuring, and the variety of the cane, but each of them is found in every cane ash. The figures given below may be regarded as average examples.

| | On 100 Cane. | On 100 parts of Ash. | | | | |
|---|---|---|---|---|---|---|
| | I. | II. | III. | IV. | V. | VI. |
| Potash .. .. .. .. | 0·153 | 23·0 | 12·56 | 25·63 | 7·66 | 38·23 |
| Soda .. .. .. .... | 0 010 | 1·5 | 5·67 | 2·26 | 6 45 | 1·30 |
| Lime .. .. .. .. .. | 0·018 | 2·7 | 6·50 | 3·24 | 12·53 | 5·19 |
| Magnesia .. .. .... | 0 014 | 2·1 | 5·08 | 3·22 | 6·61 | 5·76 |
| Ironoxide .. .. .. .. | 0·003 | 0·5 | — | — | 0·56 | 1·13 |
| Alumina .. .. .... | — | — | — | — | — | 0·25 |
| Silica .. .. .. .. .. | 0·351 | 52·8 | 56·76 | 53·42 | 43·75 | 15·70 |
| Sulphuric Acid .. .... | 0·016 | 2·4 | 2·60 | 0·53 | 16·53 | 18·47 |
| Phosphoric Acid .. .. | 0·098 | 14·7 | 10·63 | 10·78 | 5·45 | 5·27 |
| Carbon .. .. .. .... | — | — | — | — | — | 0·98 |
| Chlorine .. .. .. .. | 0·002 | 0·3 | 0·20 | 0·92 | 0·21 | 4·52 |

I. Analysis by van Lookeren Campagne & v. d. Veen.
II. ,, ,, ,, ,, ,, ,,
III. ,, Bonâme.
IV. ,, ,,
V. ,, Popp.
VI. ,, Brown and Blouin.

## CHAPTER II.

# PROPORTION AND DISTRIBUTION OF THE CONSTITUENTS OF SUGAR CANE.

The constituents of the cane mentioned in Chapter I. occur in very different proportions during the whole vegetating period of the cane, and are not to be found in the same proportion in every part of the stalk. This is not to be regarded as one homogeneous mass, but as a living organism, every part of which has to fulfil its special function, and has therefore a constitution differing from that of any other part.

According to Went's* investigations, which were modified by those of Kamerling,† we may represent the formation, transformation, transposition, and accumulation of the sugars in the cane in the following way:—

**Formation of Sugar in the Cane.**

Under the influence of light the chlorophyll-bearing tissues of the leaves form carbohydrates from water and the carbonic acid of the atmosphere, probably in the first place sucrose, this formation increasing with the intensity of the light. The sucrose is transferred to the stalk, but if more sucrose is formed than can be so transferred (as will occur in the day time), the surplus is deposited in the form of starch. This starch is, however, dissolved again at night time and then transferred to the stems in the form of glucose. The leaves contain a little fructose, too, which is formed by inversion of part of the sucrose.

**Transfer of the Sugars.**

When considering the further behaviour of the sugars one must make a distinction between those which are transported from old leaves and those from young ones. Sugars coming from an old leaf reach the stem in an already adult joint. Here the sucrose does not undergo any change, the reducing sugar is used for respiration, and is thereby converted into water and carbonic acid, so that its amount constantly diminishes without, however, disappearing entirely.

**Transformation of the formed Sugar.**

When the sugars come from a younger leaf, where in general the assimilation is more powerful and the quantity of newly-formed carbohydrate more considerable, they reach the stem at its top. Here the sucrose becomes inverted, and the more so the more rapidly the top grows This inversion increases the isotonic power of the liquid in the cell, which in its turn promotes the growth. The reducing sugar is partly employed in the formation of the fibre, while another portion reaches the top and

* *Archief voor de Java Suikerindustrie*, 1896, 605. † *Ibid*, 1904, 780.

combines with nitrogenous substances to form albuminoids, or is deposited there as starch for subsequent use.

**Disappearance of the Reducing Sugar.**

That part of the reducing sugar which remains in the top is gradually consumed during respiration, while part of it is also deposited temporarily as starch in the leaf-sheaths and in the top joints.

**Intensity of the Assimilations.**

Kamerling in Java found that in the course of a sunny forenoon the dry substance content of powerfully assimilating cane leaves increased by 15 per cent. notwithstanding that carbohydrates had been continuously transferred to the stem during that time. Taking into consideration that forenoon only, and assuming the weight of the assimilating leaves per stalk to be 300 grms., having early in the morning a dry substance content of 20 per cent., then the leaves of one cane stalk had produced at least $300 \times 0{\cdot}20 \times 0{\cdot}15 = 9$ grammes of carbohydrates.

Browne and Blouin * in Louisiana made analyses of the leaves of a young cane at night and in the morning, and from different parts of the stalk.

| | Evening Per cent. | Morning Per cent. |
|---|---|---|
| Brix | 5·77 | 5·09 |
| Sucrose | 0·94 | 0·54 |
| Glucose | 0·63 | 0·86 |
| Fructose | 0·69 | 0·77 |
| Ash | 1·42 | 1·24 |
| Free acid | 0·27 | 0·27 |
| Combined acid | 0·62 | 0·54 |
| Nitrogenous bodies | 0·15 | 0·18 |
| Gums | 0·30 | 0·17 |

We notice in the juice of the leaves a large decrease in the amount of sucrose during the night with a corresponding increase in the amount of reducing sugars. In the top joints we note an accumulation of the reducing sugars; at this point, which is the region of active growth, we have the greatest disparity between sucrose and reducing sugars, the glucose ratio being 462. In the middle joints, where the process of growth is being suspended, we observe that the reducing sugars are in the minority in comparison with the sucrose, which is henceforth stored up in the pith as reserve material. The bottom joints illustrate the same facts, only to a greater degree. Regarding the other constituents, it will be seen that the percentages of ash, free and combined acid, nitrogenous bodies, and gums all decrease from the leaves downwards to the stalk.

**Water Consumed during Growth.**

Maxwell,† in Hawaii, showed that during the whole period of the growth of the cane a supply of 147·8 parts of water is required for the formatiou of every part of dry substance.

* *Bulletin, p.* 91 *of the Louisiana Exp. Station* 12.

† *Journ. Amer. Chem. Soc.*, 1898, 469.

When we observe a single joint, from the moment at which it is formed to the period of ripeness, it will be seen that at first it contains no carbohydrates except starch. This constituent gradually becomes consumed, probably in the formation of the fibre, and during that time the joint enters into the stage in which the leaf, attached to it, begins to assimilate. Sugars collect; the joint begins to grow, the glucose and the fructose at first remain unchanged, while the sucrose becomes for the greater part inverted. A part of the invert sugar is conveyed to the younger joints in the top and another part is consumed during the growth for the formation of the fibre. At the moment that the joint is full-grown, it thus contains little sucrose and much glucose and fructose. These two sugars are consumed in the respiration process, but in the meantime new sucrose and also glucose and fructose are continuously supplied by the leaf, the former does not now become inverted any more, nor consumed by respiration, so that finally the joint contains chiefly sucrose and only a little reducing sugar.

**Composition of a single Joint during its entire vegetative Period.**

At last the joint has reached the period in which the leaf to which it belongs has ceased to assimilate and is dying off. Now the quantity of sugars in the joint only increases by the portion which flows to it from the higher parts of the cane and thus is not fixed by the higher joints. As the distance of the joint from the assimilating leaves becomes larger, owing to the death of more and more leaves, the increase in the sugar content grows less until finally we reach the point where the influx of sugar into the joint is totally stopped, but owing to assimilation by the chlorophyll of the rind, the joint gains a little sucrose, but this profit is more than counterbalanced by the loss of reducing sugar by respiration. This sugar decreases continually until in the riper joints it sinks to about 0·20 per cent. of the weight of the cane but very seldom disappears totally. In such regions where the cane is allowed to ripen fully, some joints contain no reducing sugars at all, but in practical working where some part of the cane stalk is always unripe or overripe, we are sure to find reducing sugar in every cane juice. It is evident that as soon as the point of maturity is passed, the reducing sugar content increases because of the inversion of sucrose.

Not all the joints, however, behave in this way. First of all, the bottom joints, bearing the roots, are exceptions. The roots require reducing sugar for their growth, which is conveyed to their top for the building of the tissues and the formation of albuminoids, while part of it is deposited in the root-cap as starch. This sugar is withdrawn from the joint bearing the roots, where it is formed by inversion of the sucrose. During the development of these joints the increase in sucrose, though notable, is consequently not so considerable as in the higher joints which do not have to provide for the roots. As the former become older, all the sugar which enters into them is used for the formation of roots, so that the sugar content does not further increase.

**Bottom Joints.**

A second exception may be found when cane which in the beginning has grown slowly, and therefore already contains much sugar, suddenly shoots up into a rapid growth at a time when the sky is clouded and thus assimilation by the leaves is relatively insignificant. In this case sugar can be withdrawn from the lower tops to assist the growth of upper ones.

**Joints Growing Rapidly.**

As a result of the above-mentioned facts the maximum sucrose content of a cane will for a long time be found in that joint which is on the same level with the soil, or just above the youngest root-bearing joints, and that this maximum will be found in higher joints after every banking which covers new joints with moist earth and causes development of roots and consequently consumption of sucrose. At the age of six or seven months the maximum sucrose content will be found a little higher up the stalk, and as the time of maturity approaches will rise until rather near the top. This upward movement can still go on when older joints are already overripe and decrease in sucrose content.

**Place of the Maximum Sucrose Content in the Cane.**

**Composition of the Different Joints of Cane Stalks.**

After having ascertained that canes from the same field were of similar composition, Went analysed canes from a given field joint by joint, at different periods, and tabulated the results obtained.

The Roman figures at the headings have the following signification:—

I. Number of the joints, starting at the bottom.
II. Length of the joints in centimetres.
III. Weight of the joints in grms.
IV. Per cent. sucrose in the joint.
V. Per cent. reducing sugar in the joint.
VI. Per cent. dry substance apart from sugars in the joint.
VII. Sucrose on 100 parts of the figure from VI.
VIII. Reducing sugar on 100 parts of the figure from VI.
IX. Sucrose on 100 juice.
X. Reducing sugar on 100 juice.
XI. Sum of the figures from IX. and X.

**Young Cane.** 1 stalk, 6 months of age, 1—11 bottom part, 22—30 white top.

| I. | II. | III. | IV. | V. | VI. | VII. | VIII. | IX. | X. | XI. |
|---|---|---|---|---|---|---|---|---|---|---|
| 1—10 | 8·0 | 24·5 | 6·7 | 0·26 | 25·5 | 26·3 | 1·0 | 9·4 | 0·4 | 9·8 |
| 11—15 | 9·0 | 36·0 | 9·7 | 0·4 | 16·0 | 60·6 | 2·5 | 11·6 | 0·5 | 12·1 |
| 16—18 | 10·0 | 39·5 | 9·0 | 1·0 | 13·0 | 69·2 | 7·7 | 10·4 | 1·2 | 11·6 |
| 19—20 | 8·5 | 35·5 | 6·9 | 1·9 | 12·8 | 53·9 | 14·8 | 7·9 | 2·2 | 10·1 |
| 21—30 | 13·5 | 39·0 | 2·5 | 2·3 | 12·3 | 20·3 | 18·7 | 2·8 | 2·6 | 5·4 |
| Total .. .. | 49·0 | 174·5 | 6·94 | 1·25 | — | — | — | — | — | — |

1 stalk, 7 months of age, 1—16 bottom part, 34—40 white top.

| I. | II. | III. | IV. | V. | VI. | VII. | VIII. | IX. | X. | XI. |
|---|---|---|---|---|---|---|---|---|---|---|
| 1—10 | 9·0 | 22·5 | 5·9 | 0·14 | 30·7 | 19·1 | 0·4 | 8·5 | 0·2 | 8·7 |
| 11—16 | 8·0 | 35·0 | 8 8 | 0·16 | 22·0 | 44·0 | 0·7 | 11·3 | 0·2 | 11·5 |
| 17—18 | 5·5 | 30·0 | 12·1 | 0·21 | 17·7 | 68·3 | 1·2 | 14·7 | 0·2 | 14·9 |
| 19—20 | 6·0 | 41·0 | 13·3 | 0·24 | 15·0 | 88·9 | 1·6 | 15·7 | 0·3 | 16·0 |
| 21—22 | 6·5 | 33·0 | 12·6 | 0·25 | 14·4 | 87·5 | 1·7 | 14·7 | 0·3 | 15·0 |
| 23—24 | 8·0 | 53·5 | 12·0 | 0·35 | 13·8 | 87·0 | 2·5 | 13·9 | 0·4 | 14·3 |
| 25—26 | 6·0 | 41·0 | 10·0 | 0·7 | 13·3 | 75·2 | 5·3 | 11·5 | 0·8 | 12·3 |
| 27—28 | 7·0 | 48·0 | 8·2 | 1·1 | 13·1 | 62·6 | 8·4 | 9·4 | 1·3 | 10·7 |
| 29—30 | 6·5 | 45·5 | 6·1 | 1·5 | 13·0 | 47·0 | 11·5 | 7·0 | 1·7 | 8·7 |
| 31—32 | 7·5 | 53·0 | 3·2 | 2·2 | 10·6 | 30·2 | 20·7 | 3·6 | 2·4 | 6·0 |
| 33 | 5·5 | 33·0 | 1·5 | 2·6 | 11·0 | 13·6 | 23·6 | 1·7 | 3·0 | 4·7 |
| 34—40 | 13·0 | 40·0 | 0·8 | 2·0 | 10·9 | 7·3 | 18·3 | 0·9 | 2·2 | 3·1 |
| Total .. .. | 87·5 | 475·5 | 7·86 | 1·02 | — | — | — | — | — | — |

1 stalk, 8 months of age, 1—18 bottom part, 34—41 white top.

| I. | II. | III. | IV. | V. | VI. | VII. | VIII. | IX. | X. | XI. |
|---|---|---|---|---|---|---|---|---|---|---|
| 1—13 | 10·0 | 31·0 | 6·8 | 0·21 | 23·6 | 28·8 | 0·9 | 8·9 | 0·3 | 9·2 |
| 14—16 | 8·5 | 41·0 | 11·7 | 0·4 | 16·6 | 70·5 | 2·4 | 14·0 | 0·5 | 14·5 |
| 17 | 5·5 | 29·5 | 11·8 | 0·6 | 15·3 | 77·1 | 3·9 | 14·4 | 0·7 | 15·1 |
| 18 | 6·5 | 38·5 | 11·6 | 0·7 | 14·6 | 79·4 | 4·8 | 13·6 | 0·9 | 14·5 |
| 19 | 6·0 | 36·5 | 11·2 | 0·8 | 14·6 | 76·7 | 5·5 | 13·0 | 1·0 | 14·0 |
| 20 | 5·5 | 36·0 | 11·2 | 0·9 | 14·0 | 80·0 | 6·4 | 13·0 | 1·0 | 14·0 |
| 21—22 | 7·5 | 51·0 | 11·1 | 0·9 | 13·2 | 84·1 | 6·8 | 12·8 | 1·0 | 13·8 |
| 23 | 7·0 | 48·0 | 10·8 | 1·4 | 12·5 | 86·4 | 11·2 | 12·4 | 1·6 | 14·0 |
| 24 | 8·0 | 58·0 | 10·8 | 1·5 | 12·1 | 89·3 | 12·4 | 12·3 | 1·7 | 14·0 |
| 25 | 8·0 | 57·0 | 10·7 | 1·6 | 11·6 | 92·2 | 13·8 | 12·1 | 1·8 | 13 9 |
| 26 | 10·0 | 70·0 | 10·6 | 1·9 | 10·7 | 99·1 | 17·7 | 11·9 | 2·1 | 14·0 |
| 27 | 11·5 | 80·0 | 9·8 | 2·0 | (11 0*) | (89·1) | (18·2) | (11·0) | (2·2) | (13·2) |
| 28 | 10·5 | 77·0 | 9·2 | 2·1 | 11·2 | 83·0 | 18·7 | 10·4 | 2·4 | 12·8 |
| 29 | 11·0 | 72·5 | 7·9 | 2·2 | 11·8 | 67·0 | 18·7 | 9·0 | 2·4 | 11·4 |
| 30 | 11·0 | 72·5 | 6·9 | 2·3 | 10·6 | 65·1 | 21·7 | 7·7 | 2·5 | 10·2 |
| 31 | 12·0 | 74·5 | 5·5 | 2·4 | 11·1 | 49·5 | 21·6 | 6·2 | 2·7 | 8·9 |
| 32 | 12·0 | 71·5 | 3·8 | 2·6 | 10·6 | 35·8 | 24·5 | 4·3 | 2·9 | 7·2 |
| 33 | 10·5 | 59·0 | 2·3 | 2·6 | 9·9 | 23·2 | 26·4 | 2·6 | 2·9 | 5·5 |
| 34 | 10·0 | 42·0 | 1·3 | 2·5 | 10·2 | 12·7 | 24·5 | 1·5 | 2·8 | 4·3 |
| 35—41 | 17·5 | 40·0 | 0·8 | 1·8 | 9·7 | 8·2 | 18·6 | 0·9 | 2·0 | 2·9 |
| Total .. .. | 188·5 | 1085·5 | 8·17 | 1·75 | — | — | — | — | — | — |

* The figures in brackets are not found by direct analysis but calculated by interpolation.

1 stalk, 9 months of age, 1—20 bottom part, 39—47 white top.

| I. | II. | III. | IV. | V. | VI. | VII. | VIII. | IX. | X. | XI. |
|---|---|---|---|---|---|---|---|---|---|---|
| 1—13 | 12·0 | 50·0 | 9·0 | 0·21 | 19·2 | 46·8 | 1·1 | 11·1 | 0·3 | 11·4 |
| 14—15 | 9·5 | 49·0 | 12·6 | 0·20 | 16·9 | 74·5 | 1·2 | 15·2 | 0·2 | 15·4 |
| 16 | 7·0 | 35·0 | 13·0 | 0·32 | 16·6 | 78·3 | 1·9 | 15·6 | 0·4 | 16·0 |
| 17 | 7·5 | 40·0 | 13·1 | 0·4 | 16·9 | 77·5 | 3·0 | 15·8 | 0·5 | 16·3 |
| 18 | 7·0 | 37·0 | 13·5 | 0·5 | 14·1 | 95·7 | 3·5 | 15·6 | 0·6 | 16·2 |
| 19 | 7·5 | 45·5 | 13·5 | 0·6 | 13·4 | 100·7 | 4·5 | 15·6 | 0·7 | 16·3 |
| 20 | 6·5 | 42·0 | 13·5 | 0·6 | 13·8 | 97·8 | 4·3 | 15·6 | 0·7 | 16·3 |
| 21—22 | 9·5 | 59·0 | 13·5 | 0·5 | 14·0 | 97·4 | 3·6 | 15·6 | 0·6 | 16·2 |
| 23 | 7·5 | 52·5 | 13·5 | 0·8 | 12·3 | 109·0 | 6·5 | 15·4 | 0·9 | 16·3 |
| 24 | 9·0 | 66·5 | 13·5 | 1·1 | 11·7 | 115·4 | 9·4 | 15·3 | 1·2 | 16·5 |
| 25 | 10·0 | 76·0 | 13·5 | 1·2 | 10·6 | 127·3 | 11·3 | 15·1 | 1·3 | 16·4 |
| 26 | 10·0 | 76·5 | 13·5 | 1·2 | 10·7 | 126·2 | 11·2 | 15·1 | 1·3 | 16·4 |
| 27 | 11·5 | 86·5 | 13·7 | 1·2 | 10·5 | 130·5 | 11·4 | 15·3 | 1·3 | 16·6 |
| 28 | 11·0 | 83·0 | 13·4 | 1·3 | 10·1 | 132·7 | 12·9 | 14·9 | 1·4 | 16·3 |
| 29 | 12·0 | 88·0 | 13·0 | 1·4 | 10·3 | 126·2 | 13·6 | 14·5 | 1·6 | 16·1 |
| 30 | 10·5 | 78·5 | 12·4 | 1·5 | 10·6 | 117·0 | 14·1 | 13·9 | 1·7 | 15·6 |
| 31 | 11·0 | 75·5 | 11·9 | 1·5 | 12·2 | 97·5 | 12·3 | 13·5 | 1·7 | 15·2 |
| 32 | 12·0 | 77·0 | 11·3 | 1·5 | 11·9 | 95·0 | 12·6 | 12·8 | 1·7 | 14·5 |
| 33 | 11·5 | 71·0 | 11·4 | 1·7 | 10·9 | 104·6 | 15·6 | 12·8 | 1·9 | 14·7 |
| 34 | 9·5 | 61·0 | 9·8 | 1·8 | 11·8 | 83·0 | 15·3 | 11·1 | 2·0 | 13·2 |
| 35 | 9·5 | 56·5 | 8·9 | 1·9 | 12·2 | 72·8 | 15·6 | 10·1 | 2·2 | 12·3 |
| 36 | 11·0 | 64·0 | 7·0 | 2·1 | 11·8 | 59·3 | 17·8 | 7·9 | 2·4 | 10·3 |
| 37 | 11·0 | 63·0 | 5·0 | 2·4 | 10·9 | 45·9 | 22·0 | 5·6 | 2·7 | 8·3 |
| 38 | 11·0 | 57·5 | 3·4 | 2·4 | 10·1 | 33·7 | 23·8 | 3·8 | 2·7 | 6·5 |
| 39 | 10·0 | 43·5 | 1·9 | 2·2 | 10·3 | 18·4 | 21·4 | 2·1 | 2·4 | 4·5 |
| 40—47 | 18·0 | 42·0 | 0·8 | 1·9 | 9·8 | 8·2 | 19·4 | 0·9 | 2·1 | 3·0 |
| Total .. .. | 262·5 | 1576·0 | 11·01 | 1·31 | — | — | — | — | — | — |

The bottom part of the cane is woody and contains little sugar; the sugar content rises rapidly in the joints above ground and there possesses the maximum sucrose content of the whole cane. When, by the operation of banking, these joints are covered with moist earth, the roots sprout and withdraw sugar from them, so that the maximum sugar content is now to be found a little higher up. Then we find quite a series of joints with high sucrose contents, and after these a decrease upwards, which is the more marked when the growth is vigorous. The maximum reducing sugar content is to be found in the upper joints, rising in a cane of slow growth and falling somewhat lower in the case of a rapid growth.

Generally speaking one may conclude that a joint contains its maximum reducing sugar content at the period of its most vigorous growth or a short time after. This reducing sugar has come from inversion of sucrose, which is clearly shown by the relative proportions of the three sugars—sucrose, glucose, fructose, which in slowly growing tops are as = 4 : 2 : 1, and in rapidly growing ones = 0·8 : 1 : 1.

**Ripe Cane.** The distribution of the constituents in fully ripe canes is given by Went as follows :—

CANE PLANTED IN JULY AND ANALYSED THE NEXT YEAR.

| 25th April. | | | | 23rd May. | | | | 29th May. | | | | 30th June. | | | | 6th July. | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| No. of the joint. | Weight. | Sucrose. | Red. Sugar. | No. of the joint. | Weight. | Sucrose. | Red. Sugar. | No. of the joint. | Weight. | Sucrose. | Red. Sugar. | No. of the joint. | Weight. | Sucrose. | Red. Sugar. | No. of the joint. | Weight. | Sucrose. | Red. Sugar. |
| 1 | 91·5 | 13·7 | 0·5 | 1 | 136·5 | 12·3 | 0·5 | 1 | 141·5 | 10·6 | 0·7 | 1 | 13·5 | 8·2 | 0·55 | 1 | 72·0 | 12·1 | 0·6 |
| 2 | 75·0 | 13·3 | 0·55 | 2 | 87·0 | 12·8 | 0·6 | 2 | 116·0 | 11·4 | 0·7 | 2 | 61·5 | 9·3 | 0·6 | 2 | 91·0 | 13·0 | 0·5 |
| 3 | 54·5 | 13·4 | 0·55 | 3 | 75·0 | 11·9 | 0·6 | 3 | 118·0 | 12·3 | 0·7 | 3 | 75·0 | 9·7 | 0·6 | 3 | 110·0 | 13·7 | 0·6 |
| 4 | 67·0 | 13·1 | 0·55 | 4 | 74·5 | 13·6 | 0·7 | 4 | 123·0 | 12·7 | 0·7 | 4 | 80·5 | 10·5 | 0·7 | 4 | 120·0 | 14·0 | 0·5 |
| 5 | 71·5 | 11·0 | 0·7 | 5 | 81·0 | 13·7 | 0·6 | 5 | 117·5 | 12·1 | 0·5 | 5 | 78·0 | 10·8 | 0·8 | 5 | 118·0 | 14·8 | 0·5 |
| 6 | 70·5 | 11·6 | 0·6 | 6 | 80·5 | 13·9 | 0·7 | 6 | 109·0 | 13·6 | 0 6 | 6 | 83·5 | 10·6 | 0·8 | 6 | 114·5 | 14·7 | 0·45 |
| 7 | 74·0 | 12·1 | 0·6 | 7 | 80·0 | 14·2 | 0·7 | 7 | 102·5 | 13·0 | 0·8 | 7 | 78·5 | 11·6 | 0·8 | 7 | 104·5 | 15·2 | 0·4 |
| 8 | 72·0 | 13·1 | 0·6 | 8 | 70·0 | 14·5 | 0·55 | 8 | 118·0 | 14·4 | 0·6 | 8 | 81·0 | 11·5 | 0·8 | 8 | 102·0 | 15·4 | 0·4 |
| 9 | 73·5 | 13·1 | 0·55 | 9 | 69·5 | 14·6 | 0·6 | 9 | 115·0 | 13·1 | 0·7 | 9 | 78·5 | 12·0 | 0·6 | 9 | 81·5 | 15·8 | 0·33 |
| 10 | 67·0 | 13·0 | 0·55 | 10 | 75·5 | 14·7 | 0·6 | 10 | 120·5 | 14·4 | 0·5 | 10 | 80·5 | 12·0 | 0·5 | 10 | 73·0 | 16·3 | 0·33 |
| 11 | 73·0 | 13·3 | 0·8 | 11 | 80·0 | 15·0 | 0·6 | 11 | 119·0 | 15·0 | 0·5 | 11 | 69·5 | 13·0 | 0·4 | 11 | 84·5 | 16·2 | 0·35 |
| 12 | 70·5 | 13·4 | 0·8 | 12 | 79·0 | 15·1 | 0·5 | 12 | 110·5 | 15·3 | 0·4 | 12 | 70·0 | 13·0 | 0·33 | 12 | 81·5 | 16·5 | 0·34 |
| 13 | 71·0 | 13·1 | 0·8 | 13 | 81·0 | 16·0 | 0·5 | 13 | 96·5 | 15·6 | 0·35 | 13 | 74·5 | 13·0 | 0·4 | 13 | 82·0 | 16·4 | 0·30 |
| 14 | 62·0 | 13·2 | 0·9 | 14 | 83·0 | 15·9 | 0·5 | 14 | 95·5 | 16·2 | 0·4 | 14 | 77·0 | 14·0 | 0·26 | 14 | 76·0 | 17·1 | 0·29 |
| 15 | 67·5 | 13·2 | 0·8 | 15 | 84·5 | 15·3 | 0·6 | 15 | 96·0 | 16·6 | 0·35 | 15 | 69·0 | 14·0 | 0·29 | 15 | 82·5 | 17·2 | 0·29 |
| 16 | 64·5 | 13·4 | 0·8 | 16 | 80·5 | 15·4 | 0·55 | 16 | 87·5 | 16·2 | 0·34 | 16 | 73·5 | 14·0 | 0·32 | 16 | 84·5 | 17·2 | 0·24 |
| 17 | 62·5 | 13·9 | 0·9 | 17 | 75·0 | 15·5 | 0·4 | 17 | 82·5 | 16·6 | 0·32 | 17 | 72·0 | 15·1 | 0·27 | 17 | 78·5 | 17·3 | 0·25 |
| 18 | 58·5 | 13·1 | 0·8 | 18 | 68·0 | 15·6 | 0 6 | 18 | 76·5 | 16·7 | 0·4 | 18 | 79·0 | 15·3 | 0·19 | 18 | 74·0 | 17·5 | 0·26 |
| 19 | 56·5 | 13·2 | 0 8 | 19 | 65·5 | 15·4 | 0·6 | 19 | 70·0 | 16·4 | 0·5 | 19 | 76·0 | 15·7 | 0·20 | 19 | 65·5 | 17·4 | 0·27 |
| 20 | 59·0 | 12·8 | 0·9 | 20 | 62·5 | 14·9 | 0·6 | 20 | 63·5 | 15·5 | 0·45 | 20 | 73·0 | 16·6 | 0·24 | 20 | 61·0 | 17·8 | 0·26 |
| 21 | 60·5 | 12·7 | 1·1 | 21 | 62·0 | 13·6 | 0·9 | 21 | 69·5 | 17·0 | 0·32 | 21 | 70·5 | 16·9 | 0·20 | 21 | 62·5 | 17·4 | 0·24 |
| 22 | 59·0 | 12·3 | 1·1 | 22 | 58·5 | 12·8 | 0·7 | 22 | 64·0 | 17·0 | 0·4 | 22 | 67·5 | 16·7 | 0·21 | 22 | 58·0 | 17·0 | 0·23 |
| 23 | 54·5 | 12·0 | 1·2 | 23 | 44·5 | 13·0 | 0·8 | 23 | 56·5 | 16·4 | 0·4 | 23 | 65·0 | 17·1 | 0·23 | 23 | 53·5 | 17·1 | 0·24 |
| 24 | 51·0 | 11·1 | 1·3 | 24 | 33·5 | 12·3 | 0·9 | 24 | 50·0 | 16·7 | 0·34 | 24 | 57·5 | 17·4 | 0·16 | 24 | 43·0 | 16·8 | 0·28 |
| 25 | 47·0 | 10·2 | 1·5 | 25 | 27·0 | 9·9 | 1·1 | 25 | 49·5 | 16·1 | 0·34 | 25 | 58·0 | 16·7 | 0·21 | 25—26 | 64·0 | 15·7 | 0·29 |
| 26 | 41·5 | 6·8 | 2·1 | 26 | 24·5 | 8·8 | 1·1 | 25 | 45·0 | 15·3 | 0·35 | 26 | 53·5 | — | — | 27—28 | 44·0 | 13·5 | 0·27 |
| 27 | 40·0 | 6·2 | 2·4 | 27 | 21·5 | 7·5 | 1·4 | 27 | 43·5 | 14·9 | 0·45 | 27 | 47·5 | 16·1 | 0·4 | 29—30 | 37·5 | 13·0 | 0·29 |
| 28 | 35·5 | 4·0 | 2·9 | 28—30 | 57·0 | 4·5 | 2·1 | 28 | 35·5 | 14·5 | 0·5 | 28 | 41·5 | 15·6 | 0·19 | 31—33 | 43·5 | 11·6 | 0·4 |
| 29—34 | 54·5 | 2·2 | 3 9 | 31—36 | 30·5 | 2·0 | 1·3 | 29—30 | 57·0 | 12·8 | 0·5 | 29—30 | 63 5 | 14·5 | 0·17 | 34—36 | 37·0 | 9·9 | 0·6 |
| | 1805·0 | 12·00 | 0·96 | | 1947·5 | 13·39 | 0·70 | 31—32 | 39·0 | 10·3 | 0·8 | 31—32 | 48·0 | 13·5 | 0·32 | 37—45 | 43·5 | 5·7 | 0·8 |
| | | | | | | | | 33—34 | 33·5 | 7·4 | 1·0 | 33—34 | 38·5 | 12·5 | 0·34 | | 224·3 | 15·31 | 0·38 |
| | | | | | | | | 35—41 | 39·5 | 4·8 | 0·8 | 35—36 | 34·0 | 10·7 | 0·5 | | | | |
| | | | | | | | | | 2661·0 | 14·67 | 0·55 | 37—38 | 33·0 | 10·2 | 0·6 | | | | |
| | | | | | | | | | | | | 39—40 | 34·5 | 8·0 | 1·0 | | | | |
| | | | | | | | | | | | | 41—46 | 39·5 | 5·0 | 1·1 | | | | |
| | | | | | | | | | | | | | 2236·0 | 13·51 | 0·44 | | | | |

When cane is allowed to stand over in the fields a long time after it has reached its maximum sucrose content, the latter decreases irregularly. Although the normal distribution of sucrose in the cane is still faintly visible, the changes are unmistakable; we notice little sucrose in the bottom joints, a rapid rise in the higher joints, an irregular percentage in the intermediate joints, and a maximum near the top. On the whole the figures are lower; only the top has increased its sucrose content. The reducing sugar has also increased to over 0·50 per cent. in many joints of the cane; the top joints, having ripened, do not contain so much. But, on the whole, sucrose has become inverted in many parts of the cane.

**Overripe Cane.**

| 1. | | | | 2. | | | | 3. | | | | 4. | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| No. of the joint. | Weight. | Sucrose. | Red. Sugar. | No. of the joint. | Weight. | Sucrose. | Red. Sugar. | No. of the joint. | Weight. | Sucrose. | Red. Sugar. | No. of the joint. | Weight. | Sucrose. | Red. Sugar. |
| 1— 3 | 100·5 | 11·6 | 1·9 | 1—10 | 50·0 | 6·2 | 1·4 | 1—5 | 305·0 | 8·6 | 3·9 | 1—6 | 225·5 | 9·3 | 3·5 |
| 4 | 44·5 | 13·3 | 1·0 | 11—14 | 176·0 | 11·4 | 1·6 | 6 | 119·5 | 12·4 | 3·5 | 7 | 79·5 | 6·9 | 3·5 |
| 5 | 44·0 | 13·9 | 0·9 | 15—16 | 129·0 | 14·5 | 0·6 | 7 | 132·5 | 13·2 | 3·2 | 8 | 108·5 | 7·8 | 3·5 |
| 6 | 40·5 | 13·5 | 1·3 | 17—18 | 135·5 | 13·9 | 0·9 | 8 | 136·5 | 13·5 | 3·0 | 9 | 118·5 | 10·6 | 3·9 |
| 7 | 37·5 | 13·2 | 1·6 | 19—20 | 129·5 | 14·3 | 0·9 | 9 | 133·0 | 13·4 | 3·2 | 10 | 132·5 | 11·1 | 4·4 |
| 8— 9 | 62·0 | 13·6 | 1·6 | 21—23 | 141·5 | 14·8 | 0·7 | 10 | 132·0 | 13·7 | 3·0 | 11 | 134·0 | 12·5 | 3·8 |
| 10—11 | 49·0 | 12·6 | 1·6 | 24—26 | 121·5 | 14·9 | 0·9 | 11 | 122·0 | 14·4 | 3·0 | 12 | 133·5 | 11·8 | 3·5 |
| 12—13 | 40·0 | 11·0 | 1·6 | 27—29 | 101·5 | 14·3 | 0·7 | 12 | 112·5 | 14·6 | 2·7 | 13 | 116·5 | 11·6 | 4·8 |
| 14—16 | 53·0 | 12·8 | 1·6 | 30—32 | 122·5 | 14·2 | 0·7 | 13 | 107·0 | 15·5 | 2·3 | 14 | 123·5 | 12·5 | 4·4 |
| 17—19 | 68·0 | 13·1 | 1·5 | 33—35 | 134·5 | 13·8 | 0·9 | 14 | 99·5 | 15·6 | 2·3 | 15 | 129·0 | 12·8 | 3·9 |
| 20—21 | 64·0 | 14·4 | 1·4 | 36—38 | 139·0 | 14·1 | 0·7 | 15 | 98·5 | 15·6 | 2·3 | 16 | 128·0 | 12·9 | 4·6 |
| 22—23 | 72·0 | 14·6 | 1·6 | 39—41 | 122·5 | 15·7 | 0·55 | 16 | 99·0 | 15·3 | 2·4 | 17 | 131·5 | 13·3 | 4·1 |
| 24—25 | 52·0 | 14·1 | 1·5 | 42—44 | 84·5 | 16·2 | 0·20 | 17 | 95·5 | 14·8 | 2·5 | 18 | 135·5 | 12·8 | 3·9 |
| 26—27 | 44·0 | 14·0 | 1·4 | 45—47 | 96·0 | 16·6 | 0·21 | 18 | 88·5 | 15·2 | 2·7 | 19 | 122·0 | 12·2 | 4·0 |
| 28—31 | 81·5 | 14·8 | 1·3 | 48—52 | 63·0 | 13·3 | 0·24 | 19 | 83·5 | 15·2 | 2·5 | 20 | 117·0 | 12·5 | 3·9 |
| 32—34 | 59·5 | 14·6 | 1·1 | 53—65 | 53·5 | 11·7 | 0·27 | 20 | 81·5 | 15·5 | 2·5 | 21 | 111·0 | 12·9 | 3·7 |
| 35—38 | 81·0 | 13·2 | 1·3 | | 1800·0 | 14·00 | 9·77 | 21 | 79·0 | 15·9 | 2·3 | 22 | 108·5 | 12·2 | 3·7 |
| 39—41 | 36·5 | 9·4 | 1·6 | | | | | 22 | 80·0 | 15·6 | 2·0 | 23 | 98·0 | 11·6 | 4·0 |
| 42—48 | 37·5 | 9·1 | 1·6 | | | | | 23 | 67·0 | 15·1 | 2·4 | 24 | 99·5 | 12·0 | 3·9 |
| | 1067·0 | 13·15 | 1·46 | | | | | 24—26 | 161·0 | 15·1 | 2·4 | 25 | 97·5 | 12·1 | 3·7 |
| | | | | | | | | 27—29 | 162·5 | 15·0 | 2·5 | 26 | 85·0 | 12·1 | 3·9 |
| | | | | | | | | 30—32 | 131·0 | 14·3 | 2·3 | 27 | 80·0 | 11·9 | 3·8 |
| | | | | | | | | 33—35 | 77·5 | 12·6 | 2·7 | 28 | 77·5 | 11·2 | 3·9 |
| | | | | | | | | 36—38 | 59·5 | 12·1 | 2·7 | 29 | 66·5 | 12·1 | 4·8 |
| | | | | | | | | 39—49 | 189·5 | 10·5 | 3·5 | 30 | 67·5 | 12·6 | 3·9 |
| | | | | | | | | | 2973·0 | 13·60 | 28·4 | 31—33 | 132 0 | 12·9 | 3·9 |
| | | | | | | | | | | | | 34—36 | 103·0 | 12·9 | 3·2 |
| | | | | | | | | | | | | 37—39 | 69·0 | 12·4 | 3·0 |
| | | | | | | | | | | | | 40—57 | 128·5 | 10·6 | 2·7 |
| | | | | | | | | | | | | | 3258·5 | 11·70 | 3·95 |

**Canes Attacked by Disease or Insect Pests.**

Canes attacked by infectious diseases or by insects do not show such a regular distribution of the constituents as sound ones. Attacks of the cane borer (*Diatraea striatalis*) cause the sucrose content to fall, while that of reducing sugar remains unchanged; the same effect being brought about by attacks from woodpeckers (*Dendrocopus analis*). Attacks by the top borer (*Scirpophaga intacta*) do not seem to affect the sucrose content, but the joints attacked may become infected with the pine-apple disease or black rot, causing an increase of reducing sugars.

We give here the composition of different joints of cane attacked in some places by Borers and the Red Smut disease (caused by *Thielaviopsis aethaceticus*).

| No. of the Joint. | I. | | | II. | | |
|---|---|---|---|---|---|---|
| | Sucrose. | Reduc. Sugar. | Observations. | Sucrose. | Reduc. Sugar. | Observations. |
| 1 2 3 | 13·4 | 1·74 | | 15·6 | 1·70 | |
| 4 5 | 12·8 | 2·0 | | 15·9 | 1·33 | |
| 6 7 | 12·2 | 1·74 | | 15·1 | 1·67 | |
| 8 9 | 12·7 | 1·90 | | 14·8 | 2·0 | |
| 10 11 | 12·6 | 1·67 | Borer. | 12·0 | 3·20 | Red Smut. |
| 12 13 | 7·2 | 1·90 | Red Smut. | 11·8 | 3·07 | |
| 14 15 | 11·0 | 2·67 | | 14·4 | 2·67 | |
| 16 17 | 13·2 | 2·10 | | 12·5 | 3·33 | Red Smut. |
| 18 19 | 12·3 | 2·50 | | 16·2 | 2·10 | Borer. |
| 20 21 | 11·8 | 2·67 | | 13·6 | 2·28 | Red Smut. |
| 22 23 | 11·1 | 2·67 | | 14·2 | 2·40 | |
| 24 25 26 | 9·8 | 3·08 | Top. | | | |

**Influence of the Arrowing of Cane.**

Arrowing is disadvantageous, inasmuch as, vegetation having ceased, the cane is more liable to die off than if it had not arrowed. Arrowing does not, however, affect the composition of the cane to any extent, as may be seen from the following analyses of two canes from the same stool, one of which had arrowed and the other not.

| No. of the Joints. | Weight in grms. | Length in c.m. | Per cent. Sucrose. | Per cent. Reducing Sugar. | Per cent. Dry Substance. |
|---|---|---|---|---|---|
| I.—Arrowed. | | | | | |
| White top .. .. .. .. | 105 | 9·7 | 2·4 | 3·49 | 17·8 |
| Adult joints, 1 + 2 from | 67·5 | 11 | 8·2 | 2·84 | 22·6 |
| ,, ,, 3 + 4 the | 83·5 | 12·5 | 10·0 | 2·10 | 24·1 |
| ,, ,, 5 + 6 top. | 102 | 15 | 11·9 | 1·67 | 26·0 |

| No. of the Joints. | Weight in grms. | Length in c.m. | Per cent. Sucrose. | Per cent. Reducing Sugar. | Per cent. Dry Substance. |
|---|---|---|---|---|---|
| NOT ARROWED. | | | | | |
| White top .. .. .. .. | 90 | 8·7 | 4·2 | 3·07 | 17·8 |
| Adult joints, 1 + 2 } from | 101·5 | 12 | 5·6 | 3·75 | 18·8 |
| ,, ,, 3 + 4 } the | 112 | 12 | 8·0 | 2·84 | 20·7 |
| ,, ,, 5 + 6 } top. | 123 | 14 | 10·0 | 2·07 | 22·3 |
| II.—ARROWED. | | | | | |
| From arrow to joint No. 5 | 31·7 | 16·5 | 2·4 | 2·13 | 14·3 |
| Joints 6 + 7 | 41·8 | 10 | 4·6 | 2·56 | 20·8 |
| ,, 8 + 9 | 71·7 | 12 | 6·2 | 2·39 | 22·1 |
| ,, 10 + 11 | 76·8 | 12 | 9·4 | 2·07 | 24·0 |
| ,, 12 + 13 | 88·1 | 13 | 12·2 | 1·71 | 26·2 |
| NOT ARROWED. | | | | | |
| From vegetation point to joint 5 | 18·8 | 8 | 2·5 | 2·25 | 16·25 |
| Joints 6 + 7 | 40·6 | 9 | 4·6 | 3·66 | 18·95 |
| ,, 8 + 9 | 59·0 | 11 | 8·6 | 2·95 | 23·6 |
| ,, 10 + 11 | 80·3 | 13 | 10·8 | 2·02 | 25·4 |
| ,, 12 + 13 | 94·3 | 14·5 | 13 | 1·60 | 26·7 |

These results fully agree with those of some other experiments, in which some thousands of canes which had arrowed were cut from a field simultaneously with an equal number of canes which had not. These experiments were made on three estates; on two of which the white tops of the non-arrowing canes were cut for planting previous to grinding, while on the third the top was crushed along with the canes. Of every parcel 200 canes were measured and weighed and then crushed in a triple crushing mill, without maceration.

| | I. | | II. | | III. | |
|---|---|---|---|---|---|---|
| | Arrowed. | Not Arrowed. | Arrowed. | Not Arrowed. | Arrowed | Not Arrowed. |
| Length of the cane in metres | | | | | 2·56 m. | 2·60 m. |
| Weight of the cane with top | 1·65 kg. | 1·88 kg. | 1·17 kg. | 1·24 kg. | 1·83 kg. | 1·87 kg. |
| ,, ,, without top | — | 1·73 ,, | — | 1·09 ,, | — | 1·72 ,, |
| Polarization of the juice.... | 19·3 | 18·8 | 19·43 | 17·93 | 19·11 | 19·61 |
| Reducing sugar in the juice | 0·42 | 0·44 | — | — | 0·57 | 0·49 |
| Brix in the juice .. .... | 21·3 | 20·2 | 22·0 | 20·4 | 21·09 | 22·0 |
| Purity of the juice .. .. | 90·6 | 92·8 | 88·3 | 87·9 | 90·57 | 89·14 |
| Available sugar in the juice | 17·3 | 17·4 | 16·86 | 15·46 | 17·13 | 17·22 |
| Fibre on 100 cane .. .. | 13·2 | 13·0 | | | | |

The canes which had arrowed were slightly lighter than the corresponding canes which had not, and from which no tops were cut, but heavier than those from which the tops had been removed. The analysis of the juice reveals no great differences, except in case II. where the available sugar in the canes which had arrowed is almost 1½ per cent. more than in the canes without arrows.

Jansz* gives the following figures for juice obtained from 156 canes which had arrowed and 163 which had not:—

Arrowed canes .. 19·3 Brix .. 17·42 sucrose .. 90·25 purity.
Non-arrowing canes 19·2 ,, .. 17·40 ,, .. 90·62 ,,

No difference could therefore be detected.

When cane falls, the sucrose and the quotient of purity decrease considerably as is shown in the following analyses, made at different periods, of a fallen and a standing cane, cut from the same stool in a field of healthy Black Java cane. The joints are numbered from above downwards, after the white tops had been removed.

**Fallen Cane.**

24th March.

| Fallen Cane. | | | | | | Standing Cane. | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| No. of the Joints. | Length in c.m. | Weight in grms. | Sucrose. | Reducing Sugar. | Dry substance. | No. of the Joints. | Length in c.m. | Weight in grms. | Sucrose. | Reducing Sugar. | Dry substance. |
| 1—2 | 10 | 40·4 | 1·0 | 2·56 | 10·6 | 1—2 | 9 | 52·5 | 3·2 | 1·83 | 14·7 |
| 3—4 | 12 | 77·7 | 1·6 | 2·74 | 12·2 | 3—4 | 14 | 106·5 | 4·1 | 2·32 | 15·7 |
| 5 | 6 | 56·8 | 1·6 | 3·19 | 13·3 | 5—6 | 18·5 | 144·5 | 5·8 | 2·32 | 18·2 |
| 6 | 9 | 77·5 | 2·3 | 3·49 | 15·2 | 7—8 | 19 | 161·5 | 6·9 | 2·19 | 19·2 |
| 7 | 10 | 90·8 | 3·2 | 3·49 | 15·9 | 9—10 | 19·5 | 169·5 | 7·8 | 2·13 | 19·6 |
| 8 | 11 | 100·3 | 3·9 | 2·95 | 16·9 | 11—12 | 18·5 | 161·2 | 8·9 | 2·02 | 20·6 |
| 9 | 10 | 103·0 | 4·5 | 2·95 | 15·8 | 13—14 | 18 | 145·5 | 9·1 | 2·02 | 21·8 |
| 10 | 9 | 104·1 | 4·5 | 2·95 | 16·7 | 15—16 | 17 | 139·7 | 9·6 | 1·47 | 21·8 |
| 11 | 9 | 103·6 | 5·1 | 2·74 | 17·1 | 17—18 | 16 | 141·5 | 9·8 | 1·47 | 21·8 |
| 12 | 9 | 103·2 | 5·4 | 2·64 | 16·7 | 19—20 | 18 | 155·2 | 10·0 | 1·53 | 22·4 |
| 13—14 | 18 | 207·0 | 6 | 2·39 | 16·5 | 21—22 | 19 | 163·2 | 10·2 | 1·11 | 22·8 |
| 15—16 | 19 | 207·4 | 7 | 2·25 | 17·5 | 23—24 | 16 | 154·7 | 10·2 | 1·28 | 23·3 |
| 17—18 | 16 | 177·0 | 7·7 | 2·25 | 18·5 | Rest.. | 61 | 600 | | | |
| 19—20 | 18 | 196·0 | 7·9 | 1·74 | 18·3 | | 205·5 | 2346 | | | |
| 21—22 | 18·5 | 191·3 | 7·9 | 1·74 | 19·3 | | | | | | |
| 23—24 | 14·5 | 156·8 | 8·4 | 1·28 | 21·8 | | | | | | |
| 25 | 17 | 173·3 | 8·1 | 1·67 | 19·0 | | | | | | |
| 26 | 8 | 78·5 | 8·5 | 1·67 | 20·3 | | | | | | |
| 27 | 7 | 65·8 | 8·7 | 1·53 | 20·5 | | | | | | |
| 28 | 6·5 | 60·3 | 8·4 | 1·47 | 20·5 | | | | | | |
| 29 | 9 | 80·0 | 8·4 | 1·42 | 21·0 | | | | | | |
| 30 | 11 | 121·8 | 8·4 | 1·27 | 21·3 | | | | | | |
| | 225·0 | 2573 | | | | | | | | | |

* *Archief voor de Java Suikerindustrie*, 1896, 636.

19th April.

| Fallen Cane. | | | | | | Standing Cane. | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| No. of the Joints. | Length in c.m | Weight in grms. | Sucrose | Reducing Sugars. | Dry Substance. | No. of the Joints. | Length in c m. | Weight in grms. | Sucrose | Reducing Sugars. | Dry Substance. |
| 1—3 | 11 | 47·5 | 1·5 | 2·56 | 12·2 | 1—3 | 10 | 36·5 | 1·7 | 2·02 | 14·5 |
| 4—5 | 8·5 | 60·1 | 1·6 | 3·33 | 13·6 | 4—5 | 8 | 46·7 | 2·3 | 2·84 | 14·5 |
| 6—7 | 11·5 | 111·0 | 1·8 | 3·66 | 13·9 | 6—7 | 10 | 75·7 | 3·4 | 2·74 | 15·8 |
| 8—9 | 14 | 137·3 | 3·9 | 3·66 | 15·9 | 8—9 | 11 | 91·8 | 5·3 | 2·47 | 17·8 |
| 10—11 | 11·5 | 108·8 | 5·3 | 3·33 | 18·2 | 10—11 | 14·5 | 137·3 | 7·5 | 2·32 | 18·5 |
| 12 | 9 | 75·1 | 8·2 | 2·74 | 20·3 | 12—13 | 20 | 185·5 | 8·1 | 2·02 | 18·9 |
| 13 | 12 | 95·5 | 8·5 | 2·39 | 22·0 | 14—15 | 19 | 190·0 | 8·5 | 2·02 | 19·7 |
| 14 | 11 | 95·0 | 8·9 | 2·25 | 21·3 | 16—17 | 18·5 | 179·3 | 9·0 | 1·74 | 20·2 |
| 15 | 10·5 | 104·1 | 8·7 | 2·25 | 20·2 | 18—19 | 18 | 178·6 | 9·8 | 1·67 | 23·1 |
| 16 | 12 | 123·5 | 9 | 2·25 | 20·0 | 20—21 | 17·5 | 180·6 | 10·4 | 1·37 | 23·0 |
| 17 | 13 | 126·6 | 9·1 | 2·19 | 20·3 | 22—23 | 19·0 | 185·7 | 10·5 | 1·37 | 23·3 |
| 18 | 12 | 120 | 9·1 | 1·83 | 19·5 | 24—25 | 19·5 | 185·7 | 11·2 | 1·12 | 24·5 |
| 19 | 11 | 114·1 | 9·4 | 1·60 | 19·5 | 26—27 | 19·0 | 170·5 | 11·2 | 1·28 | 23·3 |
| 20 | 12 | 114·3 | 9·6 | 1·53 | 20·6 | 28—29 | 23 | 212·0 | 11·8 | 1·19 | 26·2 |
| 21 | 11·5 | 105·0 | 9·6 | 1·42 | 20·4 | 30—31 | 20 | 210·3 | 12·6 | 1·06 | 25·5 |
| 22 | 12·5 | 106·6 | 8·8 | 1·47 | 20·1 | 32 | 10 | 109·5 | 13 | 0·98 | 25·0 |
| 23 | 11·5 | 89·5 | 8·1 | 1·47 | 20·1 | | 254·5 | 2375·7 | | | |
| 24—25 | 23 | 208 | 9·2 | 1·83 | 20·8 | | | | | | |
| 26—27 | 21 | 295·5 | 9·8 | 1·67 | 19·8 | | | | | | |
| 28—29 | 24 | 214 | 9·8 | 1·60 | 19·7 | | | | | | |
| 30—31 | 25 | 242·3 | 10·1 | 1·42 | 20·2 | | | | | | |
| 32 | 12 | 122·0 | 10·2 | 1·37 | 21·4 | | | | | | |
| | 294·5 | 2715·8 | | | | | | | | | |

| Fallen Cane. | | | | | | Standing Cane. | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 1—4 | 20 | 108 | 2·2 | 3·33 | 14·0 | 1—4 | 9 | 49·8 | 3·1 | 3·49 | 18·2 |
| 5—7 | 15 | 121 | 4·2 | 3·84 | 16·8 | 5—7 | 8 | 53·6 | 5·8 | 2·95 | 23·0 |
| 8—10 | 16 | 134·5 | 7·0 | 3·07 | 20·0 | 8—10 | 7 | 51·5 | 8·4 | 1·53 | 22·3 |
| 11—13 | 12 | 98 | 8·45 | 2·47 | 20·7 | 11—13 | 10 | 73·7 | 12·0 | 0·96 | 25·5 |
| 14—16 | 12 | 98·5 | 10·1 | 1·87 | 21·2 | 14—16 | 12 | 98·5 | 13·9 | 0·69 | 26·0 |
| 17—18 | 12 | 123·5 | 11·7 | 1·67 | 21·6 | 17—18 | 10·5 | 91·5 | 15·2 | 0·64 | 26·8 |
| 19—20 | 12 | 109 | 12·0 | 1·28 | 22·9 | 19—20 | 12 | 112·0 | 15·1 | 0·64 | 26·3 |
| 21—22 | 17 | 153·5 | 12·3 | 1·10 | 22·5 | 21—22 | 18 | 174·2 | 15·1 | 0·64 | 26·7 |
| 23 | 12 | 102·2 | 12·1 | 1·10 | 23·5 | 23—24 | 19 | 196·5 | 15·0 | 0·76 | 25·8 |
| 24 | 11 | 101·1 | 12·0 | 1·10 | 21·6 | 25—26 | 18 | 178·7 | 15·2 | 0·76 | 26·2 |
| 25 | 11·5 | 115·8 | 12·0 | 1·10 | 22·3 | 27—28 | 19 | 202·2 | 15·7 | 0·88 | 26·3 |
| 26 | 8·5 | 87·3 | 12·3 | 0·96 | 21·3 | 29—30 | 18 | 187·0 | 15·0 | 0·76 | 26·0 |
| 27—28 | 16 | 174·2 | 12·0 | 0·93 | 21·8 | 31—32 | 15 | 141·0 | 15·0 | 0·76 | 26·9 |
| 29—30 | 16 | 160·7 | 12·0 | 0·83 | 22·9 | 33—34 | 16 | 151·3 | 15·6 | 0·69 | 27·3 |
| 31—32 | 14 | 133·1 | 12·4 | 0·76 | 21·6 | Rest. | 81 | 85·9 | | | |
| 33—34 | 15 | 122·6 | 13·2 | 0·85 | 23·5 | | 272·5 | 2621·3 | | | |
| Rest. | 93 | 89·7 | | | | | | | | | |
| | 314·0 | 2840 | | | | | | | | | |

The fallen canes are heavier and longer than the standing ones from the same stools. On the other hand, their sucrose and fibre contents are inferior, and their water and reducing sugar contents higher. These differences point to unripeness of the fallen canes, which grow more rapidly, but ripen more slowly.

**Deterioration of Cut Canes.**

Canes which are kept for a long time, either in the field or under shelter, after being cut, deteriorate very rapidly; the sucrose becomes partially inverted and the reducing sugar content consequently increases, while the weight of the cane diminishes owing to the evaporation of water. Contrary to the view generally held, the acidity of the juice does not augment during the first few days after cutting.

Müller von Czernicky* reports experiments in which a few tons of canes were completely dug out and cleaned from adhering earth, just as is the custom in Java when harvesting cane. These canes were tied into 12 bundles of 24 canes each which were preserved indoors and of which bundles two were analysed each day during six consecutive days.

| Days after the Cutting. | 0 | 1 | 2 | 3 | 4 | 5 |
|---|---|---|---|---|---|---|
| Brix .. .. .. .. .. | 21·2 | 21·6 | 21·7 | 21·8 | 22·3 | 22·5 |
| Polarization .. .. .... | 19·93 | 20·20 | 20·25 | 19·69 | 19·07 | 18·45 |
| Quotient of purity .. .. | 94·0 | 93·5 | 93·3 | 90·3 | 85·5 | 82·0 |
| Reducing sugar .. .... | 0·3 | 0·3 | 0·4 | 0·8 | 1·6 | 2·1 |
| Available sugar .. .. | 15·7 | 15·6 | 15·3 | 14·5 | 13·0 | 12·0 |
| Loss of weight in per cent. | 0 | 1·1 | 2·1 | 3·0 | 3·9 | 4·7 |

The percentage of available sugar was calculated by assuming that 100 parts of cane contain 84 parts of juice of a composition equal to that obtained by the laboratory mill; and, further, that the loss in weight is only due to evaporation of water, therefore the degrees Brix are the same for all the samples, and these are assumed to contain the same juice content. The available sugar is then calculated as being represented by the product of sucrose and quotient of purity divided by 100.

Another parcel of canes which had lain in the field for 24 hours exposed to sunshine and afterwards kept indoors, gave the following figures:—

| Days after removal from field. | 0 | 1 | 2 | 3 | 4 | 5 |
|---|---|---|---|---|---|---|
| Degrees Brix .. .. .. | 21·5 | 22·1 | 22·4 | 22·7 | 23·0 | 23·3 |
| Polarization .. .. .... | 20·35 | 20·82 | 19·36 | 18·17 | 17·77 | 17·29 |
| Quotient of purity .. .. | 94·6 | 94·2 | 86·4 | 80·0 | 77·2 | 74·2 |
| Reducing sugar .. .... | 0·2 | 0·3 | 1·0 | 1·9 | 2·3 | — |
| Available sugar .. .. | 16·2 | 16·0 | 13·5 | 11·6 | 10·8 | 9·9 |
| Loss of weight per cent.. | 0 | 2·1 | 3·3 | 4·3 | 5·4 | 6·6 |

* *Archief voor de Java Suikerindustrie*, 1900, 610.

Savornin Lohman* obtained similar results when analysing cane which had been kept indoors for 24 hours, as compared with cane that had been left in the field during that time, and with cane just brought in.

| Constituents. | Crushed immediately. | Kept in the field for 24 hours. | Kept indoors for 24 hours. |
|---|---|---|---|
| Brix .. .. .. .. .. | 17·13 | 16·48 | 16·89 |
| Polarization .. .. .... | 14·63 | 13·54 | 14·17 |
| Purity .. .. .. .. | 85·45 | 82·13 | 83·90 |
| Available sugar .. .... | 13·64 | 12·36 | 13·08 |
| Acidity .. .. .. .. | 0·190 | 0·202 | 0·200 |

When calculating the available sugar to the same figures for degrees Brix we find for the canes crushed immediately 13·64 per cent., for those kept out for 24 hours 12·88 per cent. and for those kept indoors during that time 13·27 per cent., which means a loss per 24 hours on 100 parts of juice of respectively 0·76 and 0·37, or on 100 parts of original available sugar of 5·57 and 2·71 per cent.

These figures were found in the tropics (Java) but it appears that in cooler climates this deterioration is not so rapid, as is shown in H. Pellet's† table given below. H. Pellet allowed cane to lie for a long time and analysed samples from the heap at different dates.

| Days kept. | Loss of Weight in per cent. | Spec. Gr. of the Juice. | Sucrose in 100 Juice. | Reducing Sugars on 100 Sucrose. | Quotient of Purity. |
|---|---|---|---|---|---|
| 0 | 0 | 1·0705 | 13·5 | 3·3 | 88·5 |
| 4 | 2·5 | 1·0729 | 13·4 | 4·17 | 89·2 |
| 7 | 4·3 | 1·0715 | 13·6 | 3·37 | 88·1 |
| 11 | 10·0 | 1·072 | 14·2 | 3·80 | 89·6 |
| 15 | 8·7 | 1·074 | 13·7 | 4·80 | 87·0 |
| 20 | 9·0 | 1·076 | 14·3 | 3·10 | 87·8 |
| 23 | 12·5 | 1·0765 | 13·8 | 3·70 | 86·5 |
| 25 | 15·0 | 1·081 | 14·7 | 3·75 | 87·1 |
| 27 | 18·0 | 1·084 | 14·9 | 4·50 | 85·3 |

In the comparatively hotter months of December and February the deterioration was much greater, without, however, approaching that shown in the Java instances.

* *Archief voor de Java Suikerindustrie*, 1900, 1175.
† *Etudes sur la Canne à Sucre*, page 40.

In conjunction with Went the author has made experiments † as to the practicability of preserving cut canes for a long time without their undergoing too great a change. They came to the conclusion that sound canes, kept out of contact with each other and covered with moist sheets, remain unchanged for several days. But it should be stated that these experiments were only made in the laboratory, so that the results are not applicable to ordinary conditions where canes are stacked and stored in a much rougher way.

**Preservation of Cut Canes.**

A stool of black Java cane, consisting of 10 stalks, was cut, two of the canes were inspected under the microscope and afterwards crushed; half of the remaining eight canes (portion A) were placed in a dry spot in the sunshine, while the other half (portion B) were covered with moist sheets. After 1, 2, 3, and 5 days respectively, from every one of the two portions one stalk was inspected microscopically, then crushed and the juice analysed. The microscopical inspection showed that immediately after the cutting of the cane the parenchyma cells contained much sucrose and little reducing sugar. A day later a small increase in the reducing sugar content was to be observed in the cane A but not in B. Even after two or three days the B canes remained unchanged, while in those from A a good deal of reducing sugar could be detected, chiefly in those cells which were nearest to the periphery of the cane. At the end of the fifth day this decomposition of sucrose had also reached the centre of the stalk, so that in the canes from A all the parenchyma cells contained considerable quantities of reducing sugar; in B, on the contrary, only a very slight difference from the original state could be observed. We could only detect an accumulation of reducing sugar in those cells of the B canes which were situated close to the buds and to the sprouting rootlets.

The analyses of the juice confirm the microscopical tests.

| Constituents. | Immediately after. | | After 1 day. | | After 2 days. | | After 3 days. | | After 5 days. | |
|---|---|---|---|---|---|---|---|---|---|---|
| | 1 | 2 | A | B | A | B | A | B | A | B |
| Sucrose .. .. | 20·7 | 21·8 | 21·4 | 21·0 | 21·0 | 21·0 | 20·4 | 20·0 | 19·8 | 20·0 |
| Reducing sugar.. | 0·13 | 0·12 | 0·24 | 0·22 | 0·69 | 0·35 | 1·83 | 0·38 | 2·21 | 0·68 |
| Purity.. .. .. | 93·7 | 92·0 | 94·6 | 93·3 | 92·8 | 93·1 | 86·6 | 94·8 | 84·8 | 92·2 |

Next a stool of Muntok cane consisting of nine stalks was cut; one of these was crushed and analysed immediately, four others were allowed to dry by being exposed to the sun during daytime, and the remaining four were kept moist by covering them with moist sheets. After 1, 3, 10, and 25 days, respectively, a stalk was analysed from each lot. Those which had been kept wet did not show any increase of the reducing sugar content when examined under the microscope.

† *Archief voor de Java Suikerindustrie*, 1894, 249.

The same could be said of the dried cane after a day's interval, but at the end of three days the reducing sugar increased, still more so after 10 days, and, of course, very much more at the end of the twenty-fifth day. In this connection we found all the cells living in the canes which had been kept moist, even at the end of the period of 25 days, while in the case of the dried canes a great many of the parenchyma cells, especially those at the periphery, were dead after 10 days, and no living cell could be detected at the end of the twenty-fifth day. Moreover, the stalk was internally interwoven with fungoid mycelium and contained many bacteria, and the juice had become acid. Since Muntok cane usually sprouts very slowly, the rootlets had not developed to a noticeable degree. The analyses of the juice gave the following results :—

| Date of Analysis. | | Analysis of the Juice. | | |
|---|---|---|---|---|
| | | Sucrose. | Reducing Sugars. | Quotient of Purity. |
| Crushed immediately .. | | 20·32 | 0·58 | 93·2 |
| After 1 day | moist.. | 19·69 | 0·52 | 93·31 |
| | dry .. | 20·03 | 0·57 | 93·46 |
| ,, 3 days | moist.. | 18·63 | 0·70 | 91·32 |
| | dry .. | 20·71 | 1·22 | 90·81 |
| ,, 10 ,, | moist.. | 18·93 | 0·77 | 91·89 |
| | dry .. | 17·84 | 4·88 | 77·56 |
| ,, 25 ,, | moist.. | 20·17 | 0·66 | 92·95 |
| | dry .. | 12·21 | 11·20 | 52·65 |

As long as the cells remain alive, the composition of the juice does not appreciably alter, but as soon as they die the reducing sugars increase at the cost of the sucrose. The chief cause of the death of the cells is the drying up of the cane. When we succeed in preventing the cut cane from drying (a very difficult problem in tropical countries), or from fermenting, it may be kept for a long time before the juice deteriorates. In cooler climates the cane may be kept for a long time after cutting by the practice known in Louisiana under the name of

**Windrowing.**

"windrowing." When there is cane in the field at the time that frost is expected, the standing canes are cut, thrown between rows, and covered with cane trash to keep off the frost until they can be conveyed to the mill to be crushed. Stubbs* showed that, in windrowing, much sucrose is lost by inversion, so that the practice is only resorted to in order to avoid still larger loss by frost splitting.

The following figures are given by Stubbs: cane cut 7th November, windrowed till 13th December or during 35 days, maximum temperature 25·5° C., minimum 3·3° C., average 15° C., rainfall 2·6 inches.

* *Bulletin* 37, *Louisiana Exp. Station*, page 1294.

| | Brix. | Sucrose. | Reducing Sugar. | Purity. | Glucose Quotient. | Available Sugar. |
|---|---|---|---|---|---|---|
| Analysis on 7th November | 14·09 | 10·42 | 1·94 | 73·95 | 18·6 | 7·70 |
| ,, ,, 13th December | 14·23 | 9·02 | 2·99 | 64·02 | 33·1 | 5·77 |

Cane cut November 22nd and windrowed till January 2nd, or during 42 days, maximum temperature 25° C., minimum —6° C., average 11·1° C., rainfall 4 inches.

| | Brix. | Sucrose. | Reducing Sugar. | Purity. | Glucose Quotient. | Available Sugar. |
|---|---|---|---|---|---|---|
| Analysis on 22nd November | 14·22 | 11·33 | 1·60 | 80·0 | 14·1 | 9·06 |
| ,, ,, 2nd January .. | 14·22 | 10·40 | 2·38 | 73·1 | 22·9 | 7·60 |

Browne and Blouin* ascribe this gradual falling off in sucrose content of cane which has been windrowed for any length of time to spontaneous inversion by enzymes from the upper end of the cane, for if the green tops are removed at the time of the cutting the loss of sucrose is much less. This can easily be seen in the following series of experiments which were carried out at Audubon Park in 1903. Several lots of canes were windrowed, one half of each lot having the tops removed. In all other respects the conditions of the experiments were alike. At the end of a month all tops were removed, the stalks from the different lots ground and the juices analysed with the following results:—

| | Brix. | Sucrose. | Reducing Sugar. |
|---|---|---|---|
| Lot 1. Windrowed with tops cut .. .. .. | 16·1 | 13·3 | 1·25 |
| ,, ,, ,, on .. .... | 15·9 | 12·1 | 1·85 |
| Lot 2. ,, ,, ,, cut .. .. .. | 15·8 | 12·8 | 1·22 |
| ,, ,, ,, on .. .... | 15·4 | 11·5 | 1·53 |
| Lot 3. ,, ,, ,, cut .. .. .. | 16·3 | 13·5 | 1·25 |
| ,, ,, ,, on .. .... | 16·1 | 12·6 | 1·92 |
| Lot 4. ,, ,, ,, cut .. .. .. | 16·2 | 13·7 | 1·00 |
| ,, ,, ,, on .. .... | 15·8 | 11·8 | 1·85 |
| Lot 5. ,, ,, ,, cut .. .. .. | 15·9 | 12·8 | 1·39 |
| ,, ,, ,, on .. .... | 15·0 | 10·7 | 2·17 |

* *Bulletin* 91, *Louisiana Exp. Station*, page 18.

**The Effect of Frost on Sugar Cane.**

The injurious effect of frost on sugar cane is illustrated by the following examples reported by Stubbs.* On the 27th December, the canes were exposed to a sharp frost, and on the 28th December a few canes were cut and analysed, the remainder being partly windrowed, and the balance left standing. The temperature was—7·2° C. on the 27th December, and it kept on freezing till 2nd January. The cane was cut on 15th January. Thus after 20 days (maximum temperature 25° C., average 8·4° C., rainfall 8 inches,) the analyses of the juices were as follows:—

| | Brix. | Sucrose. | Reducing Sugar. | Purity. | Available Sugar. |
|---|---|---|---|---|---|
| Analyses on 27th December | 14·91 | 11·85 | 1·42 | 79·5 | 9·42 |
| 15th January (windrowed). | 13·60 | 9·09 | 1·65 | 66·9 | 6·62 |
| 15th Jan. (left standing).. | 12·84 | 8·66 | 1·29 | 67·5 | 5·84 |

Stubbs expresses the view that the frost killed the cane cells, causing an intermixture of the different kinds of sap, which then decomposed.

**Effect of Fire on Sugar Cane.**

The deterioration of cane occasioned by the death of the cells is still more rapid after a fire. When a canefield is burnt, only the dry trash is actually consumed; but the stalk itself is overheated, with the result that the cells die and the cane perishes. During the first days after a fire only a trifling change in the composition of the juice is noticeable, so that if the burnt canes are milled at once the quality of the juice is not much inferior to that of unburnt canes. The real drawback is not the actual loss of sucrose, but the necessity of crushing the burnt canes at once, which means a large pecuniary loss if the canes are not yet ripe. In the examples given below, the sucrose and the quotient of purity are very low, but these are only indirect consequences of fire, because the planter was compelled to bring the cane to the mill much earlier, and in a less ripe condition, than would otherwise have been the case.

Usually the cane can be crushed soon enough, and then the damage is not considerable, as is shown here:—

* *Bulletin* 37, *Louisiana Exp. Station*, 1224.

| | Cane variety. | Brix. | Polarization. | Purity. |
|---|---|---|---|---|
| 1st day.. .. | Black Java. | 18·3 | 14·75 | 80·6 |
| 2nd ,, .... | | 17·8 | 14·15 | 79·5 |
| 3rd ,, .. .. | | 17·3 | 13·68 | 79·1 |
| 1st ,, .... | Black Java. | 21·4 | 19·91 | 87·7 |
| 3rd ,, .. .. | | 21·3 | 18·67 | 77·3 |
| 1st ,, .... | Seedling 247. | 14·4 | 10·95 | 76·0 |
| 2nd ,, .. .. | | 13·2 | 9·80 | 74·2 |
| 3rd ,, ... | | 12·6 | 9·18 | 72·8 |
| 1st ,, .. .. | Seedling 71. | 17·3 | 14·04 | 81·1 |
| 2nd ,, .... | | 17·2 | 13·96 | 81·2 |
| 3rd ,, .. .. | | 16·0 | 12·32 | 77·0 |

But if circumstances prevent the canes being crushed at once, the deterioration goes on rapidly and uninterruptedly. In one case a field was planted with Seedling No. 247 and, on the 30th March, a plot of six acres caught fire and was destroyed. The mill was out of order just then, and the cane could not be harvested, but in order to investigate the rapidity of the deterioration, a few canes were cut from time to time, divided into pieces and analysed. Every one of the stalks was cut into 10 pieces, and the Nos. 1, 2, 3, &c., to 10 of each cane combined, crushed and analysed. The cane was harvested on 10th and 11th April, and crushed on the 12th April, while on that same date a portion of these canes was crushed and analysed in the laboratory.

| Date. | Sample taken. | Brix. | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Top. | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 |
| March 21st | Previous to the fire . .. .. | 11·5 | 14·47 | 15·48 | 15·68 | 15·78 | 15·98 | 16·03 | 16·18 | 16·58 | 17·13 |
| ,, 31st | Burnt cane .. .. .. .... | 12·2 | 15·2 | 15·9 | 16·09 | 15·94 | 15·84 | 15·59 | 15·59 | 15·75 | 16·35 |
| ,, 31st | Unburnt, from the same field | 11·85 | 14·67 | 15·57 | 15·82 | 16·17 | 16·17 | 16·12 | 16·37 | 16·72 | 17·47 |
| April 3rd .. | Burnt cane .. .. .. .. .. | 13·04 | 15·10 | 15·65 | 15·64 | 15·39 | 14·74 | 13·99 | 13·34 | 12·8 | 14·01 |
| ,, 5th .. | ,, ,, .... .. .. .... | 12·24 | 14·64 | 15·25 | 15·04 | 14·54 | 13·84 | 13·09 | 12·53 | 12·23 | 13·74 |
| ,, 7th .. | ,, ,, .. .. .. .. .. | 14·23 | 15·62 | 16·20 | 15·75 | 15·40 | 13·69 | 13·13 | 11·67 | 10·77 | 11·97 |
| ,, 9th .. | ,, ,, .... .. .. .... | 11·86 | 14·49 | 14·80 | 14·39 | 13·59 | 12·84 | 12·09 | 11·38 | 11·28 | 12·04 |

| Date. | Sample taken. | Polarization. | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Top. | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 |
| March 21st | Previous to the fire . .. .. | 6·13 | 10·49 | 11·80 | 12·10 | 12·26 | 12·59 | 12·70 | 13·— | 13·63 | 14·47 |
| ,, 31st | Burnt cane .. .. .. .... | 7·45 | 11·57 | 12·53 | 12·78 | 12·72 | 12·67 | 12·55 | 12·61 | 13·96 | 13·06 |
| ,, 31st | Unburnt, from the same field | 6·82 | 10·82 | 12·28 | 12·75 | 13·21 | 13·21 | 13·18 | 13·72 | 14·5 | 15·57 |
| April 3rd .. | Burnt cane .. .. .. .. .. | 8·28 | 11·50 | 12·48 | 12·56 | 12·42 | 11·88 | 11·28 | 10·82 | 10·58 | 12·08 |
| ,, 5th .. | ,, ,, .... .. .. .... | 7·63 | 10·98 | 12·15 | 12·15 | 11·76 | 11·15 | 10·42 | 10·07 | 10·01 | 11·68 |
| ,, 7th .. | ,, ,, .. .. .. .. .. | 8·34 | 11·43 | 12·31 | 11·91 | 11·66 | 10·70 | 9·68 | 8·55 | 7·97 | 9·42 |
| ,, 9th .. | ,, ,, .... .. .. .... | 7·13 | 10·44 | 11·09 | 10·73 | 9·91 | 9·27 | 8·64 | 7·99 | 8·37 | 9·48 |

| Date. | Sample taken. | Purity. | | | | | | | | | | Reducing Sugar. | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Top | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | Top | 2 | 3 | 9 | 10 |
| March 21st | Previous to the fire.. .. .. | 53·3 | 72 5 | 76·2 | 77·2 | 77·7 | 78·8 | 79·2 | 80·3 | 82·2 | 81·6 | 3·21 | 2·60 | 2·24 | 1·98 | 1·60 |
| „ 31st | Burnt cane .. .. .. .... | 60·8 | 76·2 | 78·8 | 79·4 | 79·8 | 80·— | 80·5 | 80·9 | 82·9 | 85·4 | 2·40 | 1·80 | 1·54 | 1·18 | 1 04 |
| „ 31st | Unburnt, from the same field | 52·5 | 73·7 | 78·8 | 80·6 | 81·7 | 81·7 | 81·7 | 83·8 | 86·7 | 89·1 | 2·64 | 2·— | 1·90 | 1·29 | 0·94 |
| April 3rd . | Burnt cane .. .. .. .. .. | 63·5 | 76·16 | 79·54 | 80·3 | 80·7 | 80·6 | 80·6 | 81·6 | 82·6 | 86·2 | 2·38 | 1·62 | 1·60 | 1·19 | 1·08 |
| „ 5th . | „ „ .... .. .. .... | 62·3 | 75·— | 79·5 | 80·78 | 80·88 | 80·56 | 79·6 | 80·37 | 81·85 | 85·01 | 2·62 | 2·— | 1·72 | 1·17 | 0 96 |
| „ 7th . | „ „ .. .. .. .. . | 58·8 | 73·17 | 76·— | 75·6 | 75·7 | 78·16 | 73·7 | 73·26 | 74·— | 78·7 | 2·91 | 2·44 | 2·12 | 1·60 | 1·27 |
| „ 9th .. | „ „ .... .. .. ... | 60·1 | 72·05 | 74·9 | 74·56 | 72·9 | 72·2 | 71·46 | 70·2 | 74·2 | 78·7 | 2·79 | 2·10 | 2·— | 1·56 | 1·30 |

The mill juice from those canes cut on two consecutive days had the following composition:—

| | Brix. | Pol. | Purity. |
|---|---|---|---|
| Cut 10th April .. .. .. .. | 9·86 .. | 4·73 .. | 47·78 |
| „ 11th „ .... .. .... | 9·62 .. | 4·86 .. | 50·62 |

while the sample crushed in the laboratory mill gave 10·7, 6·79, 63·4.

In another instance where cane was left in the field for a long time after a fire, from April 9th until May 4th, the average weight of the canes diminished from 1·31 kg. to 1·05; and the composition of the juice, which was originally Brix 12·75, sucrose 8·60, reducing sugar 2·63, and purity 67·45, became Brix 8·78, sucrose 4·74, reducing sugar 3·29, and purity 53·99.

**Influence of the Character of the Soil on the Sucrose Content of the Cane.**

The character of the soil may influence the sucrose content of the cane. As a rule, heavy soils yield sweet canes and light soils poorer canes, but this is by no means always the case.

**Influence of Fertilizers on the Sucrose Content of the Cane.**

Fertilizers do not affect the sucrose content of cane, but may do so indirectly by retarding the vegetative period, and thus causing the cane to ripen in a longer or shorter time, thereby influencing the sucrose content only.

**Increase of Sucrose in Cane during Ripening.**

The following summary of hundreds of analyses of cane, which were made with a view to ascertaining the period of maturity, shows how the sucrose content and the purity increase as maturity proceeds.

| Date of Analysis. | Brix. | Sucrose. | Purity. | Available Sugar. | Date of Planting. |
|---|---|---|---|---|---|
| I. | | | | | |
| 27th March .. | 19·4 | 17·81 | 91·9 | 13·09 | 15th May. |
| 10th April .... | 19·7 | 18·34 | 93·1 | 13·67 | idem. |
| 20th „ .. | 20·1 | 18·77 | 93·3 | 14·01 | idem. |
| 30th „ .... | 20·1 | 18·75 | 93·2 | 13·98 | idem. |

| Date of Analysis. | Brix. | Sucrose. | Purity. | Available Sugar. | Date of Planting. |
|---|---|---|---|---|---|
| II. | | | | | |
| 27th April .. | 19·5 | 17·89 | 91·8 | 13·14 | 28th May. |
| 17th May .... | 19·9 | 18·23 | 91·6 | 13·36 | idem. |
| 27th ,, .. | 19·9 | 18·55 | 93·2 | 13·83 | idem. |
| 8th June ... | 19·9 | 18·66 | 93·7 | 14·00 | idem. |

**Decrease of Sucrose in Over-ripe Cane.**

If cane is allowed to stand too long the sucrose content and purity fall.

| | Brix. | Sucrose. | Purity. | Available Sugar. | |
|---|---|---|---|---|---|
| III. | | | | | |
| 6th May.. .. | 18·9 | 17·26 | 91·4 | 13·62 | Black Java. |
| 24th ,, .... | 19·3 | 17·76 | 92·1 | 13·09 | |
| 3rd June .. | 19·7 | 18·41 | 93·4 | 13·75 | |
| 12th ,, .... | 19·9 | 18·59 | 93·4 | 13·88 | |
| 2nd July .. | 19·3 | 17·59 | 91·7 | 12·96 | 12 months of age. |

**Influence of Rain on the Ripening of Cane.**

The sucrose content of cane diminishes after a rain shower which follows a prolonged spell of drought, because the cane absorbs water, but the quotient of purity is not affected.

| Date. | Brix. | Pol. | Purity. | |
|---|---|---|---|---|
| IV. | | | | |
| 11th June.. .. .. .. .. | 19·1 | 17·45 | 91·2 | rain |
| 21st ,, .. .. .. .... | 18·9 | 17·35 | 91·8 | |
| 2nd July.. .. .. .. .. | 19·3 | 17·94 | 92·9 | |
| 12th ,, .. .. .. .... | 19·5 | 18·30 | 93·8 | |
| 22nd ,, .. .. .. .. .. | 19·6 | 18·39 | 93·8 | |

**Influence of Nitrogenous Fertilizers on the Ripening.**

As we have already shown, a heavy manuring with nitrogenous fertilizers retards the ripening of the cane, so that sugar cane, which has not been manured, already contains its maximum sugar content at a time when heavily manured cane, planted at the same time in the same field, is still unripe.

Not fertilized.

| | Brix. | Pol. | Purity. | Available Sugar. |
|---|---|---|---|---|
| V. | | | | |
| 29th April, 9¼ months .. | 20·8 | 19·36 | 93·1 | 14·43 |
| 5th May, 9½ ,, .... | 21·2 | 19·36 | 93·6 | 14·89 |
| 15th May, 9¾ ,, .. .. | 21·2 | 19·78 | 93·3 | 14·78 |
| Fertilized with 160 lb. of ammonium sulphate per acre. | | | | |
| 5th May, 9½ months .... | 20·5 | 18·85 | 92·95 | 13·86 |
| 15th May, 9¾ ,, .. .. | 20·8 | 19·39 | 93·3 | 14·47 |
| 2nd June, 10½ ,, .... | 21·5 | 20·25 | 94·2 | 15·27 |
| Fertilized with 320 lb. of ammonium sulphate per acre. | | | | |
| 5th May, 9½ months .. .. | 19·6 | 17·71 | 90·36 | 12·80 |
| 2nd June, 10½ ,, .. .... | 20·6 | 19·24 | 93·45 | 14·39 |
| 16th June, 11 ,, .. .. | 21·0 | 19·93 | 94·90 | 15·13 |

**Period of Ripening of different Varieties.**

Some varieties ripen more quickly than others, as is illustrated by the increase in the available sugar at intervals of 10 days for different varieties

| Black Java. | Seedlings No. | | | |
|---|---|---|---|---|
| | 100 | 247 | 146 | 135 |
| VI. | | | | |
| 11·62 | 11·02 | 7·87 | 10·4 | 9·77 |
| 12·64 | 11·50 | 8·15 | 10·76 | 10·08 |
| 13·20 | 12·27 | 9·22 | 10·97 | 10·47 |
| 13·88 | 12·57 | 9·34 | 11·81 | 10·52 |
| 14·38 | 12·94 | 9·35 | 12·08 | |
| | 13·71 | 9·87 | 12·35 | |
| | 13·72 | | 12·96 | |
| | 13·54 | | | |

The increase in available sugar was for

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| Black Java .. | 2·76 | per cent. in | 50 | days or | 0·55 | per. cent. in | 10 days. |
| Seedling 100 .. | 2·52 | ,, ,, | 60 | ,, | 0·42 | ,, ,, | |
| Seedling 247 .. | 2·00 | ,, ,, | 60 | ,, | 0·33 | ,, ,, | |
| Seedling 146 .. | 2·55 | ,, ,, | 70 | ,, | 0·36 | ,, ,, | |
| Seedling 135 .. | 0·75 | ,, ,, | 40 | ,, | 0·10 | ,, ,, | |

**Influence of Climate on Sucrose Content.**

In sub-tropical countries temperature has a very marked influence on the sucrose content of the cane. This has been shown for Louisiana by Browne and Blouin, in the results of years 1903 and 1904, which were very dissimilar as regards weather conditions.

1903.

| | August 1. | September 1. | October 1. | November 1. | November 15. |
|---|---|---|---|---|---|
| Sucrose .. .. .. | 2·70 | 5·97 | 11·27 | 13·60 | 15·86 |
| Reducing sugar.... | 3·80 | 3·68 | 2·51 | 1·02 | 0·63 |
| Purity .. .. .. | 36·00 | 57·02 | 76·72 | 87·85 | 92·10 |

1904.

| | | | | | |
|---|---|---|---|---|---|
| Sucrose .. .. .. | 2·35 | 5·13 | 8·04 | 9·13 | 12·00 |
| Reducing sugar.... | 4·04 | 3·75 | 3·55 | 2·82 | 1·66 |
| Purity .. .. .. | 32·28 | 52·35 | 66·61 | 71·55 | 80·53 |

The weather conditions for the two years in question during the growing season were as follows : —

| | July. | August | September | October. | November | December. |
|---|---|---|---|---|---|---|
| Av. daily temp., F. 1903 | 82·80 | 82·10 | 77·00 | 67·90 | 58·00 | 49·00 |
| ,, ,, F. 1904 | 80·90 | 84·10 | 84·20 | 71·20 | 64·30 | 58·40 |
| Rainfall, inches, 1903 .. | 5·55 | 5·98 | 1 27 | 0·36 | 0·23 | 3·89 |
| ,, ,, 1904 .. | 6·47 | 5·75 | 3·24 | 0·75 | 1·50 | 3·02 |

The results for the two years show but little variation up to the middle of September. After this date the sucrose content of the 1903 canes increased considerably, and this increase continued until the end of the season. The average daily temperature and rainfall for the two years were also about the same during June, July, and August; for the remaining months of the year, however, the conditions were very unlike September, October, November, and December. 1903 showed far lower daily averages in temperature than the corresponding months of 1904, while it also showed a deficiency of rainfall. These conditions for 1903 were very adverse to the growth of the cane, yet hastened the ripening to an extent rarely attained in Louisiana. On the other hand the unusually warm weather of the fall of 1904, together with favouring rains, promoted the growth of canes even into December, but retarded the ripening.

In tropical countries where the average temperature does not vary much in different years, the sucrose content of the cane is not so much influenced by the temperature as by the rainfall.

Kobus* illustrated this in a table, in which he put together the inches of rainfall during the early growth of the cane and during the ripening time, and the average sucrose content of the cane in Java, and the yield of sugar on 100 cane. As cane is planted there in June-September and crushed in May-October of the following year, the figures for October-December, 1904, for instance, apply to the cane crushed in May-October, 1905.

| Year. | Sugar Extracted from 100 Cane. | Sucrose in 100 Cane. | May—October (Ripening time). | | October, November, December (Soon after planting). | |
|---|---|---|---|---|---|---|
| | | | Average Monthly Rainfall. | In Per 1000 of the Year's Total. | Rainfall in Three Months. | In Per 1000 of the Year's Total. |
| 1894 | 10·36 | | 2·16 | 127 | 23·62 | 368 |
| 1895 | 9·79 | | 4·66 | 249 | 18·74 | 340 |
| 1896 | 10·55 | | 0·74 | 51 | 17·60 | 310 |
| 1897 | 10·06 | | 1·38 | 87 | 19·37 | 339 |
| 1898 | 10·21 | | 3·46 | 185 | 27·95 | 417 |
| 1899 | 10·94 | 13·99 | 2·28 | 160 | 18·23 | 326 |
| 1900 | 9·57 | 12·26 | 4·56 | 263 | 17·72 | 288 |
| 1901 | 10·16 | 12·68 | 3·42 | 176 | 19·10 | 310 |
| 1902 | 10·77 | 13·43 | 1·10 | 67 | 11·85 | 96 |
| 1903 | 10·03 | 12·40 | 2·48 | 131 | 25·78 | 418 |
| 1904 | 10·74 | 13·04 | 3·07 | 174 | 20·59 | 347 |
| 1905 | 10·37 | 12·66 | 2·12 | 129 | 13·27 | 233 |
| 1906 | 10·04 | 12·44 | 3·11 | 184 | 26·61 | 396 |

The figures for rainfall during the ripening time are highest in 1895 and 1900, and they correspond with the lowest extraction, *i.e.*, low sucrose content and low purity; while, on the other hand, the driest ripening seasons, 1896 and 1902, correspond with high extraction. The rainfall during the ripening period is, however, not the only factor, as the rather rainy years, 1899 and 1904, also showed high extraction. Another factor is the meteorological state during the early period of growth, as cane can be fully ripe in May if an early growth is favoured by a plentiful rainfall in October. Here we find the highest figures for 1898, 1903, and 1906, and, in accordance with these, high extraction in the corresponding years 1899, 1904, and 1907, notwithstanding excessive rainfalls during the ripening periods in those years.

* *Handelingen*, 8 *Suiker Congress*, 40.

**Influence of Conditions of Cultivation on the Sucrose Content.**

When stools are allowed to ratoon, the sucrose content of the ratoon canes is generally superior to that of the plant canes, although the tonnage is apt to diminish every year, unless the field is "supplied" with new plants which cannot be counted as ratoons.

Chiquelin and Verret* report the following results of their analyses of plants and of first and second ratoons of two varieties of cane, which clearly illustrate the falling off in weight and the improvement in sucrose content.

| | | Plant. | 1st year Ratoons. | 2nd year Ratoons. |
|---|---|---|---|---|
| Striped cane. | Weight of stalk | 1894 gm. | 1262 gm. | 1042 gm. |
| | Fibre | 6·56 % | 7·45 % | 8·02 % |
| | Sucrose | 4·79 | 6·03 | 8·45 |
| | Dextrose | 2·05 | 2·27 | 1·97 |
| | Levulose | 1·60 | 1·73 | 1·64 |
| | Ash | 0·39 | 0·27 | 0·27 |
| | Acids | 0·21 | 0·18 | 0·11 |
| | Albuminoids | 0·08 | 0·06 | 0·07 |
| | Amids | 0·08 | 0·02 | 0·02 |
| | Gums | 0·06 | 0·07 | 0·08 |
| D. 74 cane. | Weight of stalk | 1575 gm. | 1497 gm. | 1163 gm. |
| | Fibre | 6·28 % | 7·12 % | 7·16 % |
| | Sucrose | 6·33 | 7·36 | 8·24 |
| | Dextrose | 1·84 | 1·65 | 1·83 |
| | Levulose | 1·35 | 1·20 | 1·12 |
| | Ash | ·40 | ·41 | ·39 |
| | Acids | ·21 | ·22 | ·18 |
| | Albuminoids | ·06 | ·04 | ·07 |
| | Amids | ·10 | ·03 | ·02 |
| | Gums | ·07 | ·07 | ·09 |

There can, of course, be no general rule, since the two succeeding crops ripen in two different years under sometimes very different circumstances. In some years a long drought retards the growth of the plant canes and, in such a year, a poor crop will be obtained, while in the following year if there is more rain the ratoons of that cane may yield a heavy return.

**Ratoons.**

It is therefore obvious that the difference in yield per acre between plant canes and the different ratoons cannot be exactly stated, but, generally speaking, the weight of cane per acre diminishes every time the canes are ratooned.

* *Bulletin* No. 91 *of the Louisiana Exp. Station.*

A couple of analyses out of many hundreds made by Francis Watts* in Antigua show the quality of the juice from plant canes and their first ratoons, the figures being obtained by analysing a few canes from every parcel.

I.—Sealy Seedling, Cassada Garden.

| | Weight Cane. Tons per acre. | Per cent. Juice. | Sucrose lbs. per gallon. | Glucose lbs. per gallon. | Non-sugar lbs. per gallon. | Total Solids lbs. per gallon. | Purity. |
|---|---|---|---|---|---|---|---|
| Plant, 1906, *a* .. .. | 14·6 | 53·2 | 2·254 | 0·033 | 0·136 | 2·423 | 93·0 |
| ,, ,, *b* .... | 12·9 | 55·5 | 2·361 | 0·027 | 0·186 | 2·361 | 90·9 |
| First Ratoons, *a* .. .. | 20·2 | 53·0 | 2·145 | 0·088 | 0·167 | 2·400 | 89·4 |
| ,, ,, *b* .... | 20·2 | 56·0 | 2·228 | 0·069 | 0·151 | 2·448 | 91·0 |

II.—Queensland Creole, Diamond.

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| Plant, 1906, *a* .. .. | 32·4 | 54·5 | 2·106 | 0·023 | 0·132 | 2·261 | 93·0 |
| ,, ,, *b* .... | 37·7 | 57·0 | 2·184 | 0·043 | 0·107 | 2·334 | 93·6 |
| First Ratoons, *a* .. .. | 16·8 | 52·0 | 1·685 | 0·063 | 0·165 | 1·913 | 88·1 |
| ,, ,, *b* .... | 16·6 | 52·0 | 1·937 | 0·112 | 0·165 | 2·214 | 87·5 |

**Variations in Sucrose Content and Purity.**

In order to show the differences in sucrose content of cane and first mill juice during the course of the grinding season, we here give the average sucrose content of cane and the Brix, sucrose, and quotient of the undiluted juice extracted during the various periods of the season, on three different estates.

| I. | | | | | II. | | | | |
|---|---|---|---|---|---|---|---|---|---|
| Period. | Sucrose in Cane. | Juice. | | | Period. | Sucrose in Cane. | Juice. | | |
| | | Brix. | Sucrose. | Quotient | | | Brix. | Sucrose. | Quotient |
| April 28—May 10.. | 13·03 | 17·85 | 15·00 | 84·03 | May 23—31 .... | 13·97 | 19·16 | 17·09 | 89·19 |
| May 11—20 ..... | 13·41 | 18·26 | 14·96 | 81·92 | June 1—10..... | 14·19 | 19·29 | 17·27 | 89·25 |
| May 21—31 ..... | 13·72 | 17·97 | 15·90 | 88·48 | June 11—20 .... | 14·44 | 19·20 | 17·14 | 89·27 |
| June 1—10 ..... | 13·65 | 18·96 | 15·87 | 83·70 | June 21—30 .... | 14·27 | 18·92 | 16·91 | 89·37 |
| June 11—20 ..... | 13·44 | 18·93 | 15·92 | 84·09 | July 1—10 .... | 15·40 | 19·53 | 18·09 | 92·62 |
| June 21—30 ..... | 13·22 | 19·19 | 16·51 | 86·03 | July 11—20 .... | 15·07 | 19·43 | 18·02 | 93·16 |
| July 1—10 ..... | 13·69 | 19·28 | 16·69 | 86·56 | July 21—31 .... | 14·40 | 19·15 | 17·43 | 91·01 |
| July 11—20 ..... | 14·30 | 19·32 | 16·85 | 87·21 | August 1—10 .. | 14·40 | 19·11 | 16·95 | 88·69 |
| July 21—31 ..... | 13·38 | 18·77 | 15·98 | 85·13 | August 11—20 .. | 12·81 | 18·90 | 16·48 | 87·19 |
| August 1—10 .... | 14·26 | 18·78 | 16·30 | 86·79 | August 21—31 .. | 15·15 | 19·71 | 17·67 | 89·64 |
| August 11—20 .... | 13·96 | 19·04 | 16·56 | 86·97 | September 1—10.. | 14·78 | 18·46 | 16·61 | 89·97 |
| August 21—31 .... | 13·74 | 18·96 | 16·22 | 85·54 | September 11—20 | 14·12 | 18·54 | — | — |
| September 1—10 .. | 14·53 | 19·54 | 16·72 | 85·60 | September 21—30 | 14·28 | 18·73 | 16·46 | 87·88 |
| Total ..... | 13·73 | 18·94 | 16·16 | 85·35 | Total ..... | 14·72 | 19·20 | 17·26 | 89·89 |

* *Sugar Cane Experiments in the Leeward Islands.* Report 1905-6, 1906-7.

III.

| | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| April 23—May 15.. | 14·74 | 19·40 | 17·56 | 90·5 | July 16—31 .... | 14·51 | 18·8 | 16·78 | 89·3 |
| May 16—31 ..... | 13·65 | 18·20 | 16·01 | 88·0 | August 1—15 .. | 13·91 | 18·5 | 15·98 | 86·4 |
| June 1—15..... | 14·15 | 19·1 | 16·45 | 86·07 | Aug. 16—Sep. 5.. | 14·12 | 19·5 | 17·28 | 88·6 |
| June 16—30 ..... | 14·72 | 19·2 | 17·10 | 89·1 | | | | | |
| July 1—15..... | 14·44 | 18·7 | 16·46 | 88·0 | Total ..... | 14·39 | 18·9 | 16·78 | 88·8 |

To illustrate the variability of the sucrose contents of cane and juice in different years, the author has chosen one factory from every sugar district in Java, and here records the average sucrose content in cane and in first mill juice, as also the average purity of the latter, for the years 1899 to 1908 inclusive.

| | 1899. | | | 1900. | | | 1901. | | | 1902. | | | 1903. | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Sucrose in Cane. | Juice. | | Sucrose in Cane. | Juice. | | Sucrose in Cane. | Juice. | | Sucrose in Cane. | Juice. | | Sucrose in Cane. | Juice. | |
| | | Sucrose. | Quotient. | | Sucrose. | Quotient. | | Sucrose. | Quotient. | | Sucrose. | Quotient | | Sucrose. | Quotient |
| Cheribon .. | 13 89 | 16·94 | 90·20 | 12·31 | 15·53 | 88·94 | 13·05 | 15 73 | 88·12 | 13·84 | 16·71 | 87·51 | 12·08 | 14·77 | 83·21 |
| Tegal .... | 15·37 | 18·46 | 92 01 | 14·02 | 16·3 | 90 13 | 13·09 | 14·79 | 84·8 | 13·08 | 15·17 | 82·61 | 13·37 | 15·77 | 85·9 |
| Pekalongan | 15·26 | 17·88 | 91·65 | 13·11 | 15·45 | 88·0 | 12·14 | 14·5 | 83·7 | 14·74 | 17·24 | 89·4 | 12·58 | 15 33 | 84·7 |
| Semarang.. | 13·52 | 17·75 | 91·10 | 12·49 | 14·31 | 87·2 | — | — | — | — | — | — | 12·29 | 14·24 | 84·76 |
| Japara .... | 14 33 | 16·82 | 88 9 | 13·85 | 16·62 | 87·7 | 13·66 | 16 64 | 88·74 | 14·69 | 18 01 | 87·17 | 13·72 | 16·85 | 85·97 |
| Banjoemas. | 11·70 | 14·75 | 87·48 | 10·40 | 12·88 | 83·79 | 11·50 | 14·60 | 86·8 | 12·16 | 14·89 | 86·36 | 10·62 | 13·01 | 81·0 |
| Djokdja... | 12·89 | 15·72 | 93·0 | 11 33 | 12·65 | 84·5 | 12·2 | 15·5 | 87·37 | 13·29 | 18·04 | 88·6 | 11·79 | 14·43 | 82·88 |
| Solo .. .. | 14·34 | 16·43 | 89·8 | 12·31 | 14·82 | 86·1 | 14·15 | 17·29 | 88·4 | 14·61 | 17·74 | 88·3 | 13·67 | 16·12 | 83·3 |
| Madioen . | 14·66 | 17·59 | 92·33 | 12·84 | 15·51 | 88·78 | 12·78 | 15·88 | 87·88 | 14·08 | 17·03 | 87·15 | — | — | — |
| Kediri .... | 13·84 | 16·57 | 88·09 | 11·27 | 13·27 | 82·17 | 11·4 | 13 61 | 79·6 | 13·03 | 15·90 | 87·4 | 11·26 | 13·4 | 80·24 |
| Soerabaja .. | 13·40 | 15·92 | 87·3 | 10·35 | 12·52 | 82·2 | 11·38 | 13 74 | 84·55 | 12·12 | 14·65 | 83·19 | 11·33 | 13·57 | 82·24 |
| Pasoeroean. | 15·01 | 17·35 | 90·98 | 14·35 | 16·68 | 90·16 | 14·97 | 17·53 | 92·11 | — | — | — | 14·99 | 17·65 | 89·1 |
| Probolinggo | 14·51 | 18·02 | 90·92 | 13·78 | 16·22 | 89·12 | 13·61 | 15·72 | 87·82 | 13·35 | 16·53 | 84·0 | 12·88 | 15·39 | 83·76 |
| Besoeki .... | — | — | — | — | — | — | — | — | — | — | — | — | 10·05 | 11·82 | 78·9 |

| | 1904. | | | 1905. | | | 1906. | | | 1907. | | | 1908. | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Cheribon .. | 13·38 | 16·57 | 88·51 | 13·46 | 16·58 | 87·12 | 12·85 | 15·62 | 86·44 | 13·50 | 16·76 | 88 06 | 11·84 | 14·72 | 85·13 |
| Tegal .... | 14·53 | 17·30 | 88·3 | 12·80 | 15·43 | 84·78 | 12·59 | 15·27 | 85·25 | 14·28 | 17·29 | 88·97 | 13 31 | 16·31 | 89·47 |
| Pekalongan | 13·44 | 16·27 | 87·38 | 13·22 | 16·00 | 87·11 | 12·87 | 16·00 | 88·16 | 13·75 | 16·78 | 90 17 | 12·34 | 15·26 | 88 10 |
| Semarang. | 13·42 | 15·76 | 88·39 | 13 63 | 16·21 | 88·05 | 12 55 | 15·07 | 83·80 | 13·89 | 17·00 | 87·58 | 13·00 | 15·76 | 86·36 |
| Japara .... | 14·20 | 17·48 | 88·96 | 13·16 | 16·89 | 86·75 | 13·58 | 17·24 | 88·41 | 13·70 | 16·86 | 89·60 | 13·51 | 16·35 | 88·57 |
| Banjoemas. | 11·58 | 14·40 | 88·19 | 12·69 | 15·13 | 88·58 | 11·77 | 13 97 | 85·39 | — | — | — | — | — | — |
| Djokdja.... | 12·79 | 15·16 | 84·36 | 12·90 | 15·55 | 84·74 | 12·36 | 14·81 | 83·67 | 14·16 | 17·28 | 87·98 | 13·09 | 15·79 | 86·23 |
| Solo .. .. | 14·33 | 17·18 | 89·0 | 13·72 | 16·38 | 84·70 | 13·60 | 16·47 | 86·36 | 14·15 | 17·30 | 90·20 | 13·12 | 16·09 | 87·97 |
| Madioen . | 15 18 | 17 71 | 89·41 | 14·27 | 16·86 | 89·11 | 14·12 | 16·58 | 88·24 | 14·52 | 17·06 | 88·60 | 13·88 | 16·54 | 86·90 |
| Kediri ... | 12·43 | 14·73 | 84·32 | 11·37 | 13·96 | 81·64 | 10·64 | 13·92 | 84·16 | 11·52 | 14·54 | 83·18 | 11·70 | 14·42 | 83·40 |
| Soerabaja .. | 11·45 | 14·12 | 84·19 | 10·98 | 13·89 | 83·08 | 10·51 | 13·52 | 82·74 | 12·11 | 15·20 | 86·91 | 10·83 | 13 74 | 84·08 |
| Pasoeroean. | 14·68 | 17·34 | 90·08 | 14·00 | 16·52 | 90·17 | 14·09 | 16·64 | 88·46 | 13·64 | 17·04 | 88·10 | 12 44 | 16·19 | 89·00 |
| Probolinggo | 13·16 | 18·81 | 85·69 | 12·71 | 15·56 | 86·07 | 12·13 | 14·90 | 83·61 | 13·30 | 16·15 | 87·49 | 12·52 | 15·85 | 87·13 |
| Besoeki .... | 10·76 | 12·68 | 80·7 | 11·42 | 13·47 | 82·00 | 9·94 | 12·13 | 79·30 | 10·10 | 12·06 | 79·90 | 10·63 | 12·76 | 81·80 |

It is extremely difficult to obtain figures for the average sucrose content of the canes in certain countries. Figures can be quoted, but they relate chiefly to the results of a single estate in one season, and cannot be considered as representing averages.

One must therefore abstain from repeating here the various items of information to be found scattered throughout the literature of the subject, and only mention that in countries where the period of vegetation is shortened by frost, the sucrose content generally remains low, because the great bulk of the cane is harvested before maturity. In countries, having little rainfall, as in some parts of Hawaii and Peru, when the cane sometimes stands 20 months or more and is liable to become partially dry when fully ripe, the sucrose content may rise as high as 16 per cent. and that of the reducing sugar fall very low. In the broad belt of sugar-producing countries with a hot and moist climate, viz., Central America, the Antilles, and Java, the sucrose content is fairly constant and, though occasionally exceptionally high or low, this percentage may be put down as 13 per cent. in the cane when it reaches the mill.

**Sucrose Content of Cane in various Countries.**

It is to be hoped that the exaggerated percentage of 18-20 per cent. which we so often find copied from one handbook to another will, at last, be replaced by the more reasonable limits of 12-15 per cent.

**Mutual Proportion of the three Sugars in Cane.**

The proportion in which the three sugars occur in the cane largely depends on the age of the different parts of the cane.

We gave 0·73 per cent. sucrose and 0·60 per cent. reducing sugar, consisting of glucose and fructose, in cane leaves which are actively assimilating.

The following figures are for cane at different periods of development.

| | Sucrose. | Glucose. | Fructose. |
|---|---|---|---|
| White tops of cane 6 months old .... .. | 1·02 | 1·24 | 1·25 |
| ,, ,, 9 ,, ,, .. .... | 1·90 | 1·30 | 0·70 |
| Bottom joints ,, 12 ,, ,, .... .. | 16·50 | 0·60 | 0·20 |

The fructose content decreases gradually, but even in the juice of the riper joints its presence may always be detected by means of the reaction mentioned on page 40 with ammonium molybdate and acetic acid.

The table of Browne given below also shows that the amount of reducing sugar and especially of fructose decreases as ripening proceeds.

**Decrease in the Reducing Sugar content during Ripening.**

| Constituents. | 9th September. | 23rd September. | 7th October. | 21st October | 4th November. | 18th November | 28th November. |
|---|---|---|---|---|---|---|---|
| Brix.. .. .. .. .. .. | 10·47 | 11·52 | 14·69 | 14·70 | 15·48 | 17·22 | 19·45 |
| Sucrose .. .. .. .... | 5·97 | 7·32 | 11·27 | 11·60 | 13·60 | 15·86 | 18·29 |
| Glucose .. .. .. .. .. | 2·15 | 2·00 | 1·58 | 1·20 | 0·46 | 0·36 | 0·21 |
| Fructose .. .. .. .... | 1·53 | 1·55 | 0·93 | 0·97 | 0·56 | 0·27 | 0·04 |
| Ash .. .. .. .. .. .. | 0·36 | 0·32 | 0·32 | 0·35 | 0·36 | 0·32 | 0·32 |
| Free Acid .. .. .. ... | 0·07 | 0·07 | 0·09 | 0·08 | 0·10 | 0·07 | 0·06 |
| Combined Acid .. .. .. | 0·08 | 0·08 | 0·09 | 0·10 | 0·10 | 0·09 | 0·17 |
| Albuminoids .. .. .... | 0·09 | 0·08 | 0·08 | 0·08 | 0·08 | 0·10 | 0·18 |
| Gum.. .. .. .. .. .. | 0·08 | 0·07 | 0·07 | 0·05 | 0·17 | 0·14 | 0·18 |
| Purity.. .. .. .. .... | 57·02 | 63·54 | 76·72 | 78·91 | 87·85 | 92·10 | 94·04 |
| Glucose Co-efficients .. .. | 61·64 | 48·50 | 22·27 | 18·70 | 7·50 | 3·97 | 1·37 |

| Character of the Juice. | Brix. | Polarization before Inversion. | Polarization after Inversion. | Calculated Rotation due to Sucrose. | Rotation of the Reducing Sugar. | Reducing Sugar. | Specific Rotation of the Reducing Sugar. | Sucrose. | Glucose. | Fructose. |
|---|---|---|---|---|---|---|---|---|---|---|
| Bourbon Cane — Bottom piece.. | 18·8 | 71·9 | — 21·1 | 72·6 | — 0·66 | 1·27 | — 13·5 | 17·57 | 0·76 | 0·51 |
| Bourbon Cane — Middle piece .. | 18·5 | 69·3 | — 20·9 | 70·5 | — 1·17 | 1·34 | — 22·5 | 17·06 | 0·73 | 0·61 |
| Bourbon Cane — Top piece .... | 14·3 | 44·8 | — 14·7 | 46·5 | — 1·70 | 2·30 | — 19·4 | 11·44 | 1·17 | 1·03 |
| Black Java .. — Bottom piece. | 16·3 | 60·3 | — 18·0 | 61·2 | — 0·90 | 1·42 | — 16·0 | 14·93 | 0·72 | 0·60 |
| Black Java .. — Middle piece . | 15·4 | 54·3 | — 16·5 | 55·2 | — 0 90 | 1·61 | — 14·2 | 13·52 | 0·97 | 0·64 |
| Black Java .. — Top piece . | 11·7 | 30·2 | — 11·0 | 32·2 | — 2·00 | 2·30 | — 20·3 | 8·02 | 1·27 | 1·03 |
| ,, ,, .. — Bottom half | 14·4 | 48·0 | — 13·9 | 48·3 | — 0·30 | 2·20 | — 3·9 | 11·88 | 1·43 | 0·77 |
| ,, ,, .. — Top half.. . | 11·2 | 29·8 | — 10·5 | 31·5 | — 1·70 | 3·02 | — 1·4 | 7·85 | 2·11 | 0·91 |
| ,, ,, .. — Bottom half .. | 19·9 | 75·3 | — 21·9 | 75·9 | — 0·6 | 0·67 | — 21·2 | 18·25 | 0·34 | 0·31 |
| ,, ,, .. — Top half.. .. | 17·5 | 65·3 | — 19·7 | 66·4 | — 1·1 | 1·05 | — 27·2 | 16·13 | 0·57 | 0·58 |
| ,, ,, .. — Bottom half .. | 22·5 | 86·2 | — 24·2 | 86·2 | 0 | 0·50 | 0 | 20·52 | 0·35 | 0·15 |
| ,, ,, .. — Top half.. . | 22·76 | 93·0 | — 26·0 | 92·9 | + 0·1 | 0·35 | + 0·7 | 22·11 | 0·25 | 0·10 |
| ,, ,, .. — Bottom half .. | 22·07 | 89·3 | — 25·0 | 89·3 | 0 | 0·37 | 0 | 21·30 | 0·25 | 0·12 |
| ,, ,, .. — Top half ... | 22·73 | 92·4 | — 25·6 | 92·2 | + 0·2 | 0·37 | + 14 | 21·94 | 0·30 | 0·07 |
| ,, ,, .. — Bottom piece.. | 19·94 | 78·21 | — 22·0 | 78·3 | — 0·1 | 0·83 | — 3 | 18·82 | 0·54 | 0·29 |
| ,, ,, .. — Middle piece .. | 20·38 | 80·3 | — 23·0 | 80·7 | — 0·4 | 0·55 | — 20 | 19·41 | 0·30 | 0·25 |
| ,, ,, .. — Top piece .... | 21·05 | 82·7 | — 23·6 | 83·0 | — 0·3 | 0·64 | — 12 | 19·88 | 0·46 | 0·18 |
| ,, ,, First Mill Juice | 18·87 | 69·5 | — 19·8 | 69·8 | — 0·3 | 1·10 | — 7 | 16·86 | 0·71 | 0·39 |
| ,, ,, Mixed Juice .. | 18·40 | 67·2 | — 18·7 | 67·1 | — 0·1 | 1·46 | — 2 | 16·22 | 1·01 | 0·45 |
| ,, ,, ,, ,, .. | 19·7 | 70·5 | — 20·0 | 70·7 | — 0·2 | 1·16 | — 1·4 | 16·92 | 0·81 | 0·35 |
| ,, ,, ,, ,, .. | 19·2 | 67·5 | — 19·25 | 67·7 | — 0·2 | 1·61 | — 3·6 | 16·21 | 1·06 | 0·56 |
| ,, ,, ,, ,, .. | 16·0 | 48·5 | — 13·9 | 48·7 | — 0·2 | 2·39 | — 2· | 11·88 | 1·59 | 0·80 |
| ,, ,, ,, ,, .. | 18·8 | 61·0 | — 17·8 | 61·6 | — 0·6 | 2·00 | — 7·1 | 14·88 | 1·30 | 0·70 |
| ,, ,, ,, ,, .. | 19·1 | 69·1 | — 18·9 | 68·8 | — 0·3 | 1·42 | — 5·4 | 16·56 | 0·92 | 0·50 |
| ,, ,, ,, ,, .. | 19·3 | 68·4 | — 16·2 | 69·2 | — 0·8 | 2·27 | — 9·1 | 16·66 | 1·45 | 0·82 |
| ,, ,, ,, ,, .. | 19·0 | 70·0 | — 20·0 | 70·3 | — 0·3 | 1·22 | — 6·4 | 16·92 | 0·79 | 0·43 |
| ,, ,, ,, ,, .. | 17·9 | 64·5 | — 18·5 | 65·0 | — 0·5 | 1·46 | — 8·6 | 15·62 | 0·92 | 0·54 |

**Rotatory Power of Reducing Sugars in Cane.**

The second table on the previous page gives analytical data concerning the juice of ripe and nearly-ripe cane, in which the rotatory power of the reducing sugar is expressed in degrees Ventzke.

From the rotations at a temperature of 28°, glucose = 80 and fructose = — 134, the proportions of the two reducing sugars may be calculated from their total quantity and the rotation of the mixture.

As a rule the ripest cane contains the smallest percentage of fructose, though it must here be explained that these figures possess only relative values, since the slightest deviation in temperature or error in polariscopic reading is sufficient, when so small a quantity is under consideration, to influence the result considerably. A difference in the reading 0·1° is sufficient, in some cases, to change a positive rotatory power into a negative one, so that the figures given in the preceding table should not be regarded as rigidly accurate.

**Fibre Content of different Parts of Cane.**

The fibre content in the different parts of the cane varies considerably; not only do the bottom joints contain more fibre than the middle and upper joints, as is clearly shown by the many analyses on page 58 and following, but in the same joint the fibre content varies considerably according to the larger or smaller percentage of fibrovascular bundles and parenchyma cells which are present in the different parts.

Winter* separated, as far as possible, the hard fibrovascular bundles from the soft pith of the cane, which had previously been skinned, and ascertained the weight of both constituents; but admitted that the separation was by no means complete.

| | | Sucrose. | Fibre. | Juice. | Sucrose in 100 Juice. |
|---|---|---|---|---|---|
| 1 | Fibrovascular bundles .. .. | 15·63 | 14·22 | 85·78 | 18·22 |
| | Parenchyma cells .. .... | 18·88 | 5·00 | 95·00 | 19·87 |
| 2 | Fibrovascular bundles .. .. | 15·47 | 11·75 | 88·25 | 17·53 |
| | Parenchyma cells .. .... | 19·29 | 4·50 | 95·50 | 20·20 |
| 3 | Fibrovascular bundles .. .. | 14·04 | 12·28 | 87·72 | 16·01 |
| | Parenchyma cells .. .... | 17·40 | 4·41 | 95·59 | 18·20 |
| 4 | Fibrovascular bundles .. .. | 9·83 | 9·00 | 91·00 | 10·80 |
| | Parenchyma cells .. .... | 11·11 | 4·20 | 95·80 | 11·60 |
| 5 | Fibrovascular bundles .. .. | 14·54 | 9·17 | 90·83 | 16·01 |
| | Parenchyma cells .. .... | 16·15 | 4·00 | 96·00 | 16·82 |

In a second series with ripe and unripe cane the purity of the juice was also determined.

* *Mededeelingen van het Proefstation, Kagok I., page 30.*

| | Cane. | | | | | | Juice. | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | Sucrose. | Fibre. | Juice. | Dry. Substance | Non-sugar. | Purity. | Sucrose. | Dry Substance. | Non-sugar. | Purity. |
| Fibrovascular bundles.. | 14·54 | 9·17 | 90·83 | 26·50 | 2·79 | 54·87 | 16·01 | 19·08 | 3·07 | 83·91 |
| Parenchyma .. .... | 16·15 | 4·00 | 90·60 | 21·82 | 1·67 | 74·01 | 16·82 | 18·56 | 1·74 | 90·63 |
| Fibrovascular bundles.. | 11·55 | 9·61 | 90·39 | 23·06 | 1·90 | 50·09 | 12·78 | 14 88 | 2·10 | 85·89 |
| Parenchyma .. ... | 12·24 | 4·25 | 95·75 | 18·04 | 1·55 | 67·85 | 12·78 | 14·40 | 1·62 | 88·75 |
| Fibrovascular bundles.. | 10·98 | 12·57 | 87·43 | 25 64 | 2·09 | 42·82 | 12·56 | 14·95 | 2·39 | 84·01 |
| Parenchyma .. ... | 11·00 | 5·76 | 94·24 | 18·70 | 1·94 | 58 82 | 11·67 | 13·73 | 2·06 | 85·00 |

**Sugar-free Sap in the Fibro-vascular Bundles.**

Winter remarks that the fibrovascular part of the cane contains so little sugar that he firmly believes the bundles to be totally sugar-free, which is in accordance with the fact mentioned by the same investigator that the watery drops which ooze out from the ends of cane stalks while these are being crushed in the factory mills contain no sugar and only a little starch and salts. Went observes that although the fibrovascular bundles are the channels through which the sugar is conducted through the stalk, yet it is not altogether surprising that they contain a sugarless sap, since the sugars are transported in so dilute a solution as not to be detected by the usual analysis.

**Difference in Fibre Content of a single Joint.**

Winter found a great difference in composition in various parts of the same joint, according as they belonged to the rind, the periphery, or the centre of the internode. The joints of a cane were cut from the stalk with a saw and the nodes separated, so that only the internodes were examined. A cylindrical piece 1·5 cm. diameter was cut out from the middle of each internode in a longitudinal direction by means of a cork-borer to represent the "centre." The remainder, after being pealed, served to represent the "periphery," whilst the "rind" was cut into pieces not over 1 mm. thick with scissors.

| | | Sucrose. | Fibre. | Juice. | Sucrose in Juice. |
|---|---|---|---|---|---|
| 1 | Centre .. .. .. .... | 17·6 | 4·46 | 95·54 | 18·42 |
| | Periphery .. .. .. .. | 18·5 | 6·15 | 92·85 | 19·92 |
| | Rind .. .. .. .. .... | 9·6 | 25·31 | 74·69 | 12·85 |
| 2 | Centre .. .. .. .. .. | 15·0 | 3·57 | 96·43 | 15·56 |
| | Periphery .. .. .. .... | 14·6 | 6·45 | 93·55 | 15·61 |
| | Rind .. .. .. .. .. | — | 29·29 | 70·71 | — |

| | | Sucrose. | Fibre. | Juice. | Sucrose in Juice. |
|---|---|---|---|---|---|
| 3 | Centre | 19·2 | 4·72 | 95·28 | 20·15 |
| | Periphery | 19·0 | 9·29 | 90·71 | 20·95 |
| | Rind | 5·99 | 41·75 | 58·25 | 10·28 |
| 4 | Centre | 17·6 | 4·68 | 95·32 | 18·46 |
| | Periphery | 17·1 | 8·60 | 91·40 | 18·71 |
| | Rind | 5·21 | 46·11 | 53·89 | 9·67 |

Winter also ascertained the fibre content of the internodes and the nodes belonging to them, in which case the piece of the stalk 1·5 cm. on either side of the axis of the leaf was considered to be the node.

| | | Sucrose. | Fibre. | Juice. | Sucrose in Juice. |
|---|---|---|---|---|---|
| 1 | Nodes | 15·5 | 16·46 | 83·54 | 18·55 |
| | Internodes | 17·5 | 11·69 | 88·31 | 19·82 |
| 2 | Nodes | 15·7 | 11·62 | 88·38 | 17·76 |
| | Internodes | 17·5 | 9·15 | 90·85 | 19·26 |
| 3 | Nodes | 13·8 | 17·77 | 82·23 | 16·78 |
| | Internodes | 16·9 | 11·02 | 88·98 | 18·99 |
| 4 | Nodes | 17·5 | 18·31 | 81·69 | 21·42 |
| | Internodes | 19·1 | 11·52 | 88·48 | 21·59 |
| 5 | Nodes | 13·4 | 16·51 | 83·49 | 16·05 |
| | Internodes | 16·4 | 10·02 | 89·98 | 18·23 |

Beeson* investigated the composition of nodes and joints of the different parts of the stalk, and obtained the following data :—

| | | Dry Substance. | Sucrose. | Reducing Sugar. | Purity. | Non-sugar. | Glucose ratio. | Fibre in Cane. |
|---|---|---|---|---|---|---|---|---|
| Top end | Nodes | 15·3 | 11·30 | 0·10 | 73·9 | 3·82 | 1·60 | 15·86 |
| | Internodes | 16·9 | 14·30 | 1·25 | 84·6 | 1·35 | 8·37 | 8·60 |
| Middle | Nodes | 16·7 | 13·7 | 0·07 | 82·0 | 2·90 | 0·57 | 15·90 |
| | Internodes | 17·7 | 16·0 | 0·98 | 90·4 | 0·72 | 6·13 | 8·00 |
| Bottom end | Nodes | 15·7 | 12·8 | 0·15 | 81·5 | 2·75 | 1·17 | 18·28 |
| | Internodes | 17·7 | 16·4 | 0·61 | 92·6 | 0·69 | 3·78 | 8·00 |

* *Bull. Assoc. Chim. Sucr. et Dist.*, 1895-6, 362.

The nodes and the rind contain the largest percentage of fibre and the older parts still more than the younger ones. Winter as well as Beeson, Bonâme,* and Pellet,† observe that the hardest parts yield the least pure juice, while the latter two also mention that the hard rind contains the most impure juice, rich in reducing sugars and darkly coloured.

**Fibre Content of Pith Bundles and Rind.**

According to Browne,‡ the pith occupies 70 per cent. of the volume of the cane, but its fibre content is so little that it does not amount to more than 25 per cent. of the whole quantity of dry fibre. A mechanical separation of the tissues of the cane from one another gave the following percentage composition. The analyses were performed upon a mature stalk of the Louisiana Purple cane.

| | Pith. Per cent. | Bundles. Per cent | Rind. Per cent. |
|---|---|---|---|
| Whole cane (three analyses).... | 2·39 .... | 1·81 .... | 5·51 |
| Dry fibre .. .. .. .. .. | 24·66 .... | 18·60 .... | 56·74 |

The following figures were found for some very common Java varieties: —

| | Cheribon. | No. 36. | No. 100. | No. 139. | No. 247. |
|---|---|---|---|---|---|
| Pith.. .. .. .. . | 25·6 | 20·0 | 22·82 | 20·0 | 20·0 |
| Bundles .. ... | 31·5 | 26·5 | 25·37 | 35·97 | 26·1 |
| Rind .. .. .. | 42·9 | 53·5 | 51·81 | 44·03 | 53·9 |

From a physical point of view these fibres show a distinctly-marked difference. The rind and the bundles are woody, while the pith consists of thin-walled cells. The difference in water-absorbing power is very considerable. The bundles only absorb five times their weight of water, whereas the fibre from the parenchyma absorbs as much as 30 times its own weight.

As the chemical composition of the fibre does not vary very much, it is mainly the physical condition of same which accounts for the observed differences in water-absorbing power of the bagasse of the different cane varieties.

| Constituents. | Fibre from the Pith | Fibre from the Bundles. | Fibre from the Rind. |
|---|---|---|---|
| Cellulose.. .. .. .. .. | 49·00 | 50·00 | 51·05 |
| Pentosan .. .. .. .... | 32·04 | 28·67 | 26·93 |
| Albuminoids.. .. .. .. | 1·94 | 2·00 | 2·19 |
| Cane wax and fat .. .... | 0·41 | 0·72 | 0·98 |
| Ash.. .. .. .. .. .. | 1·68 | 3·58 | 1·64 |
| Lignin (by difference) .... | 14·93 | 15·03 | 17·17 |

* *La Canne à Sucre.* † *Etudes sur la Canne à Sucre*, 30.
‡ *Archief voor de Java Suikerindustrie*, 1904, 584.

According to van Lookeren Campagne,* cane juice contains an average of 0·07 per cent. of albuminoids, consisting of two kinds, one of which is coagulated by heat and lime, and amounts to 80 per cent. of the total quantity, whilst the other remains in solution after liming and boiling. Furthermore, the juice contains the same amount of other nitrogenous bodies, chiefly amido acids. After heavy crushing, the resulting juice contains more albumen than after moderate crushing.

**Nitrogenous Bodies.**

The nitrogenous bodies are not distributed evenly throughout the stalk, the work of Beeson having shown a larger accumulation of albuminoids in the nodes and of amids in the internodes, which accounts for the higher percentage of albuminoids in juice extracted by heavy mills, since these are able to crush the hard nodes also.

| | Albuminoids Nitrogen. | Amid Nitrogen. | Total Nitrogen. |
|---|---|---|---|
| Nodes | 0·1778 | 0·0051 | 0·1829 |
| Internodes | 0 0559 | 0·0258 | 0·0817 |

**Mineral Matters (Ash) in the Cane.**

As already stated the mineral matter occupies a rather subordinate place in the quantitative composition of the cane. We have already given the inorganic constituents of the whole cane and of the fibre, and now include in table on next page those of the juices of different cane varieties.

**Lime.**

Calcium phosphate is very prominent among the inorganic elements of the cane, but the amount of lime otherwise combined is not considerable.

**Magnesia.**

Magnesia is also always present but only in small quantity.

**Soda.**

Sodium oxide occurs in very small proportions even when the cane is grown on salt land. If the soil contains sodium sulphate, or sodium, calcium,

**Potash.**

or magnesium chlorides, these salts act on the potash silicates of the soil in such a manner that potash is liberated and rendered available to the cane. This is the reason why potash is about the only alkali base in the cane, unless the soil is quite destitute of potash and rich in sodium chloride. In such a case the cane is compelled to absorb sodium chloride, but does not thrive and soon dies. The juice of such a cane gave the following figures calculated on the weight of 460 grms. of cane and on 100 parts of the cane.

| | | | | |
|---|---|---|---|---|
| Soda | 1·016 | grm. | in per cent. | 0·221 |
| Potash | 0·199 | ,, | ,, ,, | 0·043 |
| Lime | 0·358 | ,, | ,, ,, | 0·078 |
| Magnesia | 0·146 | ,, | ,, ,, | 0·032 |
| Sulphuric acid | 0·680 | ,, | ,, ,, | 0·147 |
| Phosphoric acid | 0·107 | ,, | ,, ,, | 0·023 |
| Carbonic acid | 0·295 | ,, | ,, ,, | 0·064 |
| Silica | 0·915 | ,, | ,, ,, | 0·199 |
| Chlorine | 0·870 | ,, | ,, ,, | 0·190 |
| | 4·586 | ,, | ,, ,, | 0·997 |
| Abstract oxygen equivalent to chlorine | 0·196 | ,, | ,, ,, | 0·043 |
| | 4·390 | ,, | ,, ,, | 0·954 |

* *Archief voor de Java Suikerindustrie*, 1899, 757.

| Estate. | Variety. | On 100 Juice. | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Brix. | Quotient of Purity. | Ash. | Silica. | Ferric and Calcium Phosphate. | Lime otherwise combined. | Magnesia. | Sulphuric Acid. | Chlorine. | Potash. | Carbonic Acid in the Ash. | Carbonic Acid on 100 Ash. |
| Wonopringgo . | Cheribon.. . | 21·51 | 95·12 | 0·110 | 0·022 | 0·034 | 0·010 | — | 0·005 | 0·003 | 0·016 | 0·011 | 10·0 |
| ,, . | No. 247 .. .. | 18·87 | 85·10 | 0 131 | 0·035 | 0·041 | 0·013 | — | 0·009 | 0·002 | 0·016 | 0·011 | 8·4 |
| Djatibarang . | Cheribon.. .. | 20·4 | 88·82 | 0·189 | 0·034 | 0·045 | 0·004 | — | 0·038 | 0·009 | 0·057 | 0·011 | 5·3 |
| Asembagoes .. | No. 105 .. . | 13·7 | 74·38 | 0·767 | 0·040 | 0·035 | 0·012 | 0·013 | 0·097 | 0·172 | 0·403 | 0·025 | 3·2 |
| ,, . | Louziers .. .. | 19 0 | 83·10 | 0·521 | 0·025 | 0·059 | 0·00 | 0·019 | 0·052 | 0·111 | 0·241 | 0·016 | 3·1 |
| ,, . | No. 105 .. .. | 16·6 | 81·02 | 0·776 | 0·027 | 0·018 | 0·007 | 0·009 | 0·101 | 0·181 | 0·428 | 0·034 | 4·4 |
| Kentjong .. .. | G. Z. A. .. .. | 15·3 | 79·02 | 0 230 | 0·039 | 0·052 | 0·007 | 0 008 | 0·039 | 0·005 | 0·056 | 0·001 | 0·3 |
| Poerwodadi . | —— | 16 5 | 83·6 | 0·342 | 0·156 | 0·081 | 0·006 | 0·002 | 0·038 | 0·017 | 0·113 | 0·015 | 4·4 |
| ,, . | —— | 17·7 | 91·90 | 0·297 | 0·022 | 0·032 | 0·003 | 0 005 | 0·061 | 0·025 | 0·140 | 0·017 | 5·7 |
| Karangsoewoeng | —— | 20·53 | 83·05 | 1·027 | 0·042 | 0·190 | 0·022 | 0·018 | 0·163 | 0·153 | 0·474 | 0·064 | 6·2 |
| Balapoelang . | —— | 19·6 | 87·96 | 0 514 | 0·064 | 0·025 | 0·010 | 0·016 | 0·025 | 0·040 | 0·222 | 0·080 | 15·6 |
| ,, . | —— | 16·5 | 81·52 | 0·748 | 0·080 | 0·070 | 0·007 | 0·008 | 0·092 | 0·065 | 0·342 | 0·084 | 11·2 |
| Bantoel .. . | —— | — | — | 0·454 | 0·020 | 0·037 | 0 051 | 0·012 | 0·037 | 0·036 | 0·170 | 0·040 | 8·9 |
| Waroe.. .. .. | No. 247 .. .. | 15·37 | 81·86 | 0·540 | 0·042 | 0·084 | 0·008 | 0·003 | 0·064 | 0 100 | 0·233 | 0·017 | 3·2 |
| ,, .. .. . | No. 247 .. .. | 18·18 | 87·36 | 0·349 | 0·016 | 0·062 | 0·007 | 0·003 | 0·024 | 0·065 | 0·155 | 0·024 | 7·0 |
| Soedhono .. . | —— | 16·65 | 89·3 | 0·517 | 0·271 | 0·101 | 0 016 | 0·003 | 0·018 | 0·008 | 0·067 | 0·025 | 4·8 |
| Kanigoro .. .. | No. 139 .. .. | 15·68 | 82·5 | 0·252 | 0·057 | 0·052 | 0·002 | 0·006 | 0·029 | 0·011 | 0 076 | 0·018 | 7·2 |
| ,, .. . | Striped Preanger .. | 16·16 | 85·5 | 0·275 | 0·043 | 0·044 | 0·003 | 0·004 | 0·043 | 0·017 | 0·094 | 0·016 | 6·8 |
| ,, .. .. | No. 36 .. .. | 14·52 | 80 7 | 0·472 | 0·138 | 0·088 | 0·009 | 0·010 | 0·061 | 0·021 | 0·115 | 0·018 | 3·8 |
| ,, .. .. | Manilla .. .. | 15·18 | 80·8 | 0 420 | 0·029 | 0·100 | 0·009 | 0·010 | 0·076 | 0·008 | 0·125 | 0·043 | 10·2 |
| ,, .. . | No. 71 .. .. | 12·15 | 72·3 | 0·200 | 0·042 | 0·057 | 0·004 | 0·002 | 0·040 | 0·00 | 0·045 | 0·009 | 4·5 |
| ,, .. .. | Cheribon.. .. | 18·21 | 89·7 | 0·164 | 0·014 | 0·018 | 0·005 | 0·007 | 0·024 | 0·003 | 0·052 | 0·021 | 12·8 |
| ,, .. .. | No. 86 .. .. | 16·34 | 85 1 | 0·285 | 0·017 | 0·036 | 0·010 | 0·016 | 0·057 | 0·017 | 0·099 | 0·034 | 11·9 |
| ,, .. .. | No. 142 .. .. | 14·11 | 80·1 | 0·187 | 0 016 | 0·048 | 0·006 | 0·015 | 0·026 | 0·006 | 0·054 | 0·008 | 4·3 |
| ,, .. .. | No. 33a .. .. | 14·35 | 79·3 | 0·224 | 0·026 | 0·054 | 0·010 | 0·022 | 0·038 | 0·006 | 0 060 | 0 012 | 5·0 |
| ,, .. .. | No. 33a .. .. | 14·35 | 79·7 | 0·238 | 0·029 | 0·058 | 0·011 | 0·022 | 0·039 | 0·008 | 0·062 | 0·013 | 5·5 |
| ,, .. .. | No. 146 .. .. | 13·10 | 76·2 | 0·458 | 0·017 | 0 040 | 0·004 | 0·005 | 0·074 | 0·010 | 0 243 | 0·058 | 12·7 |
| ,, .. .. | No. 135 .. .. | 11·98 | 69·6 | 0·222 | 0·033 | 0·047 | 0·005 | 0·002 | 0·036 | 0·013 | 0·068 | 0·018 | 8·5 |
| ,, .. .. | No. 247 .. .. | 14·96 | 75·2 | 0·195 | 0·027 | 0·054 | 0·006 | 0·005 | 0·030 | 0·012 | 0·051 | 0·014 | 7·2 |
| ,, .. .. | No. 247 .. .. | 14·27 | 72·0 | 0·214 | 0·032 | 0·059 | 0·005 | 0·005 | 0·034 | 0·005 | 0·057 | 0·017 | 7·9 |

If, on the contrary, the soil contains sufficient potash, the cane does not absorb soda, but much more potash than if no sodium salts had been present. Lime and magnesia salts also have the property of liberating potash from the silicates and putting it at the disposal of the cane. The cane absorbs those salts also, but more lime than magnesia. Cane first requires potash, and only in a minor degree lime and magnesia, and, lastly, soda in the total absence of other alkali salts, but this latter constituent can by no means take the place of potash in the nutrition of the cane. When much potash is absorbed, the cane ash contains also much chlorine and sulphate, as that base is chiefly combined with inorganic salts in the juice. The figure for organic acids combined with bases, which is indicated by that for carbonic acid in the juice, does not rise accordingly.

**Inorganic Acids.**

**Influence of Soil on Quantity of Inorganic Constituents.**

The quantity and quality of the inorganic constituents of the cane depend largely on the character of the soil. A salt soil causes much more inorganic substance to enter into the composition of the cane than a non-saline one, as is clearly shown by the following analyses of cane planted in pots and watered with dilute solutions of various salts. The figures represent milligrammes of every constituent on 100 grms. of juice.

| | I. | II. | III. | IV. | V. | VI. | VII. | VIII. | IX. | X. | XI. | XII. |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| NaCl. ..Grms. | — | — | — | 128·7 | 128·7 | 257·4 | 386·0 | 257·4 | — | — | — | — |
| $MgCl_2$ ..Grms. | 120 | 240 | — | — | — | — | — | — | — | — | — | — |
| $CaCl_2$ ..Grms. | — | — | — | — | — | — | — | — | — | 211·0 | 315·0 | — |
| Insoluble Ash.. | 147·9 | 147·0 | 80·5 | 75·1 | 100·7 | 88·5 | 148·2 | 113·0 | 72·9 | 106·5 | 102·0 | 68·0 |
| Soluble Ash .. | 296·5 | 408·6 | 178·0 | 342·5 | 410·1 | 500·2 | 626·6 | — | 279·5 | 499·4 | 633·5 | 277·7 |
| Total Ash .... | 444·4 | 555·6 | 258·5 | 417·6 | 510·8 | 588·7 | 774·8 | — | 352·4 | 605·9 | 735·5 | 345·7 |
| $SiO_2$.. .. .. | 38·0 | 32·2 | 27·2 | 20·2 | 27·6 | 22·8 | 42·8 | — | 35·0 | 26·4 | 28·6 | 28·4 |
| $CO_2$ .. .... | 23·2 | 20·8 | 32·5 | 25·1 | 27·8 | 11·8 | 24·6 | — | 43·4 | 16·2 | 16·8 | 26·8 |
| $P_2O_5$ .. .. | 59·7 | 51·3 | 48·0 | 47·5 | 47·4 | 39·8 | 52·8 | — | 28·9 | 31·9 | 16·4 | 59·4 |
| $F_2O_3+Al_2O_3$ | 15·4 | 11·5 | 7·5 | 2·9 | 9·8 | 5·9 | 13·6 | 9·5 | 7·4 | 5·8 | 8·6 | 3·7 |
| CaO .. ... | 21·9 | 22·9 | 14·0 | 45·6 | 19·3 | 18·4 | 27·0 | 22·4 | 14·5 | 31·1 | 33·6 | 12·3 |
| MgO.. .. .. | 24·3 | 31·8 | 9·6 | 13·2 | 14·8 | 15·8 | 22·0 | 16·2 | 7·0 | 12·5 | 13·5 | 8·8 |
| $Cl_2$ .. .... | 97·0 | 161·7 | 8·4 | 85·9 | 114·0 | 182·1 | 296·6 | * | 28·1 | 198·9 | 272·1 | 10·5 |
| $SO_3$.. .. .. | 8·9 | 8·8 | 5·6 | 19·2 | 23·6 | 20·9 | 27·3 | — | 18·1 | 21·1 | 17·0 | 46·0 |
| $K_2O$ .. .... | 176·2 | 251·0 | 103·2 | 191·8 | 243·8 | 284·2 | 299·7 | — | 173·1 | 305·9 | 384·3 | 147·1 |
| $Na_2O$ .. .. | 1·7 | None | 4·4 | 15·6 | 8·4 | 28·0 | 56·1 | — | 3·2 | 1·0 | 5·9 | 5·1 |

**Influence of Cane's Age on its Ash-content.**

Bonâme's† investigations proved that the same cane possesses varying amounts of inorganic substances in the different periods of its growth. The dates of the analyses of whole canes, deprived of the leaves, are recorded here.

| Constituents. | 6th May. | 6th June. | 6th July. | 6th August. | 6th Sept. | 6th October. |
|---|---|---|---|---|---|---|
| Silica .. .. .. .. .. .. | 0·805 | 1·373 | 0·824 | 0·913 | 0·830 | 1·024 |
| Chlorine .. .. .. .. .... | 0·074 | 0·092 | 0·018 | 0·010 | 0·007 | 0·012 |
| Sulphuric Acid . .. .. .. | 0·135 | 0·260 | 0·121 | 0·167 | 0·142 | 0·173 |
| Phosphoric Acid .. .. .... | 0·104 | 0·155 | 0·078 | 0·075 | 0·070 | 0·110 |
| Lime .. .. .. .. . .. | 0·115 | 0·205 | 0·115 | 0·136 | 0·127 | 0·158 |
| Magnesia.. .. .. .. ... | 0·154 | 0·255 | 0·170 | 0·158 | 0·141 | 0·133 |
| Potash.. .. .. .. .. .. | 0·687 | 0·803 | 0·250 | 0·208 | 0·181 | 0·387 |
| Soda.. .. .. .. .. ... | 0·026 | 0·017 | 0·008 | 0·006 | 0·008 | 0·011 |
| Iron Oxide.. .. .. .. .. | 0·046 | 0·050 | 0·020 | 0·029 | 0·015 | 0·024 |
| Total .. .. .. .. | 2·146 | 3·210 | 1·604 | 1·702 | 1·521 | 2·032 |

* The soluble part of the ash of No. VIII. was lost during analysis.

† *Rapport Annuel de la Station Agronomique de l'Ile de Maurice*, 1896.

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It also follows from the analyses of Browne, on page 83, that if the ash-content of cane is not high it does not decrease much during ripening. A certain decrease will always take place, since, during the ripening process, potash, soda, and chlorine are transferred from the cane into the leaves, therefore the green tops contain the largest amount of ash of any parts of the cane.

Browne mentions the analyses of unripe cane, which he divided into sections, crushed, and analysed the juice.

| Constituents. | Green Tops cut in the Morning. | Green Tops cut in the Evening. | Green Top Joints. | Middle Joints. | Bottom Joints. |
|---|---|---|---|---|---|
| Brix | 5·09 | 5·77 | 5·06 | 8·79 | 12·65 |
| Sucrose | 0·54 | 0·94 | 0·76 | 4·12 | 8·93 |
| Glucose | 0·86 | 0·63 | 1·81 | 2·07 | 1·83 |
| Fructose | 0·77 | 0·69 | 1·70 | 1·82 | 1·23 |
| Ash | 1·24 | 1·42 | 0·50 | 0·37 | 0·33 |
| Free Acids | 0·27 | 0·27 | 0·18 | 0·09 | 0·07 |
| Combined Acids | 0·54 | 0·62 | 0·36 | 0·18 | 0·14 |
| Albuminoids | 0·18 | 0·15 | 0·07 | 0·08 | 0·08 |
| Gums | 0·17 | 0·30 | 0·15 | 0·11 | 0·05 |
| Purity | 10·60 | 16·29 | 15·02 | 46·87 | 70·59 |
| Glucose ratio | 301·85 | 137·23 | 461·84 | 94·41 | 34·26 |

The author has also analysed juice of ripe and unripe canes, which he divided into three parts, crushed, and analysed the juices, as under:—

| Constituents. | Unripe Cane from a field with high purity. | | | Ripe Cane from a field with high purity. | | | Ripe Cane from a field with low purity. | | |
|---|---|---|---|---|---|---|---|---|---|
| | Top. | Middle. | Bottom. | Top. | Middle. | Bottom. | Top. | Middle. | Bottom. |
| Brix | 11·9 | 13·5 | 14·2 | 20·3 | 20·48 | 20·37 | 17·8 | 19·6 | 19·3 |
| Sucrose | 7·47 | 10·44 | 11·57 | 18·96 | 19·51 | 19·30 | 15·05 | 17·91 | 17·52 |
| Reducing sugars | 3·40 | 2·34 | 2·06 | 0·50 | 0·36 | 0·35 | not determined. | | |
| Quotient of purity | 59·8 | 74·0 | 78·1 | 93·4 | 95·25 | 95·20 | 84·55 | 91·38 | 90·78 |
| Ash | not determined. | | | 0·208 | 0·141 | 0·158 | 0·300 | 0·257 | 0·278 |
| Soluble ash | | | | 0·111 | 0·067 | 0·081 | 0·194 | 0·193 | 0·206 |
| Potash | 0·119 | 0·073 | 0·052 | 0·063 | 0·038 | 0·047 | 0·106 | 0·104 | 0·109 |
| Sulphuric acid | not determined. | | | 0·027 | 0·017 | 0·020 | 0·078 | 0·077 | 0·081 |
| Chlorine | | | | 0·020 | 0·012 | 0·016 | 0·008 | 0·010 | 0·011 |
| Carbonic acid in the ash. | | | | 0·002 | 0·001 | 0·001 | 0·002 | 0·003 | 0·003 |

**Changes in the Quantity and Quality of the Inorganic Matter of Cane during Ripening.**

The variations in the inorganic constituents during the ripening are shown by the following series of cane juices, from the same fields, which were extracted and analysed at various stages of maturity:—

POERWODADI ESTATE.

| Cheribon (Black Java) Cane, planted July 1st, 1905. | | | | | Seedling No. 247, planted July 15th, 1905 | | | | |
|---|---|---|---|---|---|---|---|---|---|
| Constituents. | Date of the Analyses. | | | | | | | | |
| | 9th Feb., 1906. | 9th March, 1906. | 11th April, 1906. | 20th June, 1906. | 9th Feb., 1906. | 9th March. 1906. | 11th April, 1906. | 20th June, 1906. | 11th July, 1906. |
| Brix | 13·2 | 15·0 | 16·3 | 19·50 | 12·0 | 13·0 | 13·7 | 16·50 | 16·90 |
| Polarization | 8·75 | 11·80 | 13·74 | 18·32 | 7·13 | 9·04 | 10·46 | 14·04 | 14·90 |
| Purity | 66·3 | 78·70 | 84·30 | 93·95 | 59·4 | 69·5 | 76·25 | 85·61 | 88·20 |
| Reducing sugars | 3·8 | 2·32 | 1·18 | 0·49 | 3·30 | 2·92 | 1·51 | 1·52 | 1·17 |
| Ash | 0·207 | 0·205 | 0·224 | 0·262 | 0·299 | 0·304 | 0·207 | 0·216 | 0·273 |
| Insoluble ash | 0·068 | 0·099 | 0·092 | 0·063 | 0·089 | 0·133 | 0·075 | 0·081 | 0·075 |
| Soluble ash | 0·139 | 0·106 | 0·152 | 0·199 | 0·210 | 0·171 | 0·132 | 0·135 | 0·198 |
| Potash | 0·079 | 0·052 | 0·072 | 0·115 | 0·120 | 0·091 | 0·077 | 0·072 | 0·114 |
| Carbonic acid in the ash | 0·009 | — | — | 0·016 | 0·017 | 0·008 | — | — | 0·012 |
| Sulphuric acid | 0·040 | 0·036 | 0·056 | 0·062 | 0·052 | 0·048 | 0·059 | 0·060 | 0·063 |
| Chlorine | 0·009 | 0·010 | 0·004 | 0·008 | 0·022 | 0·016 | 0·007 | 0·003 | 0·012 |

ASEMBAGOES ESTATE.

| Cheribon (Black Java) Cane, planted June 25th, 1905. | | | | | Seedling No. 247 planted September 20th, 1905. | | | | |
|---|---|---|---|---|---|---|---|---|---|
| Constituents. | Date of the Analyses. | | | | | | | | |
| | 14th Febr., 1906. | 14th March, 1906 | 23rd April, 1906. | 14th May, 1906. | 13th June, 1906. | 14th Febr.. 1906. | 24th April, 1906. | 14th May, 1906. | 15th June, 1906. |
| Brix | 14·1 | 16·30 | 20·0 | 20·50 | 21·20 | 8·90 | 12·30 | 11·90 | 14·40 |
| Polarization | 10·46 | 13·40 | 18·17 | 18·44 | 17·68 | 3·60 | 8·14 | 7·87 | 10·20 |
| Purity | 74·18 | 82·14 | 90·85 | 89·95 | 83·39 | 40·45 | 66·18 | 66·13 | 70·83 |
| Reducing sugars | 0·81 | 1·14 | 0·62 | 0·82 | — | 3·10 | 3·09 | 1·64 | 3·07 |
| Ash | 0·825 | 0·519 | 0·475 | 0·426 | 0·585 | 0·648 | 0·522 | 0·530 | 0·641 |
| Insoluble ash | 0·202 | 0·079 | 0·073 | 0·072 | 0·083 | 0·086 | 0·153 | 0·070 | 0·096 |
| Soluble ash | 0·623 | 0·440 | 0·402 | 0·354 | 0·502 | 0·526 | 0·369 | 0·460 | 0·545 |
| Potash | 0·362 | 0·255 | 0·219 | 0·186 | 0·285 | 0·341 | 0·230 | 0·270 | 0·326 |
| Carbonic acid in the ash | 0·030 | 0·028 | — | — | 0·015 | 0·022 | 0·016 | 0·002 | 0·017 |
| Sulphuric acid | 0·101 | 0·070 | 0·181 | 0·144 | 0·181 | 0·101 | 0·054 | 0·126 | 0·125 |
| Chlorine | 0·136 | 0·088 | 0·003 | 0·021 | 0·030 | 0·126 | 0·103 | 0·095 | 0·103 |

KENTJONG ESTATE.

| Constituents. | Cheribon (Black Java) Cane. | | | | | | Seedling No. 100. | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Date of the Analyses. | | | | | | | | | | | |
| | 15th Febr., 1906. | 14th Mar., 1906. | 16th April. 1906. | 17th May, 1906. | 15th June, 1906. | 14th July, 1906. | 14th Febr., 1906. | 14th March, 1906. | 16th April. 1906. | 17th May, 1906. | 15th June, 1906. | 14th July, 1906. |
| Brix .. .. .. | 11·8 | 14·20 | 17·10 | 17·80 | 19·40 | 19·70 | 12·4 | 13·1 | 14·60 | 19·30 | 16·90 | 19·50 |
| Polarization .... | 7·44 | 10·66 | 15·19 | 16·18 | 17·97 | 18·70 | 7·75 | 8·90 | 11·72 | 17·29 | 15·35 | 17·86 |
| Purity.. .. .. | 63·05 | 75·0 | 88·83 | 90·90 | 92·63 | 94·92 | 62·5 | 67·94 | 80·72 | 89 59 | 90·83 | 91·59 |
| Reducing Sugars | 3·58 | 2·88 | 1·64 | 1·28 | 0·79 | 0·62 | 3·84 | 3·84 | 2·16 | 1·22 | 2·39 | 0·89 |
| Ash .. .. .... | 0·239 | 0·274 | 0·266 | 0·204 | 0·201 | 0·189 | 0·343 | 0·255 | 0·204 | 0·328 | 0·263 | 0·311 |
| Insoluble ash .. | 0·088 | 0·072 | 0·070 | 0·075 | 0·089 | 0·067 | 0·154 | 0·056 | 0·067 | 0·086 | 0·076 | 0·070 |
| Soluble ash .... | 0·151 | 0·202 | 0·190 | 0·129 | 0·112 | 0·122 | 0·189 | 0·199 | 0·137 | 0·230 | 0·187 | 0·241 |
| Potash.. .. .. | 0·082 | 0·110 | 0·103 | 0·062 | 0·064 | 0·070 | 0·092 | 0·099 | 0·074 | 0·128 | 0·109 | 0·143 |
| Carbonic acid in the ash .... | 0·005 | 0·010 | — | — | 0·006 | 0·011 | 0·016 | 0·012 | — | — | 0·017 | 0·025 |
| Sulphuric acid .. | 0·050 | 0·056 | 0·088 | 0·053 | 0·042 | 0·041 | 0·048 | 0·048 | 0·055 | 0·095 | 0·053 | 0·067 |
| Chlorine .. .... | 0·012 | 0·014 | 0·002 | 0·003 | — | — | 0·015 | 0·020 | 0·008 | 0·010 | 0·011 | 0·008 |

**Influence of Milling Power on the Quantity and Quality of Inorganic Substances in the Juice.**

The power of the mills in which the cane is crushed has a very marked influence on the ash content of the juice, inasmuch as the juice extracted by the heaviest crushing contains more, both soluble (potash ash) and insoluble (silicate ash) than juice which is extracted by light crushing. The following analyses of juice extracted in a triple crushing mill distinctly show the difference. The juice was obtained by triple crushing without maceration, and, in order to have comparable figures, the percentage of the different constituents is calculated to 100 parts of juice of the same density as the first mill juice.

| Constituents. | I. | II. | III. | I. | II. | III. | I. | II. | III. |
|---|---|---|---|---|---|---|---|---|---|
| Ash (total) .. .. .. .. | 0·247 | 0·360 | 0·376 | 0·202 | 0·251 | 0·418 | 0·602 | 0·567 | 0·661 |
| Soluble ash .. .. .... | 0·136 | 0·171 | 2·233 | 0·101 | 0·120 | 0·131 | 0·466 | 0·445 | 0·493 |
| Insoluble ash .. .. .. | 0·111 | 0·189 | 0·143 | 0·101 | 0·131 | 0·247 | 0·136 | 0·122 | 0·168 |
| Potash .. .. .. .... | 0·080 | 0·097 | 0·133 | 0·057 | 0·069 | 0·098 | 0·265 | 0·282 | 0·280 |
| Sulphuric acid .. .. .. | 0·029 | 0·041 | 0·049 | 0·025 | 0·030 | 0·042 | 0·128 | 0·099 | 0·147 |
| Chlorine .. .. .... | 0·027 | 0·034 | 0·045 | 0·018 | 0·024 | 0·033 | 0·054 | 0·025 | 0·056 |
| Carbonic acid in soluble ash | 0·004 | 0·002 | 0·007 | 0·001 | 0·001 | 0·002 | 0·022 | 0·028 | 0·015 |

**Mineral Constituents of Juice of different Cane Varieties from the same Field.**

Different cane varieties can absorb varying amounts of inorganic matter from the soil of the same field. The author has analysed the juice of Muntok and Canne Morte canes which were planted in the same field as Cheribon cane, and in almost every case found them to contain more ash than the latter, although grown in the same field and under the same conditions.

| Constituents. | Kemanglen Estate. | | Klampok Estate. | | Kalimati Estate. | | Wono-pringgo Estate. | | Modjopangoeng Estate. | | | | Remboen Estate. | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | | | | Kentos Field. | | Pakoentjen Field. | | | |
| | Muntok. | Cheribon. | Muntok. | Cheribon. | Muntok. | Cheribon. | Muntok. | Cheribon. | Fiji. | Cheri-bon. | Fiji. | Cheri-bon. | Fiji. | Cheribon. |
| Ash | 0·262 | 0·240 | 0·360 | 0·252 | 0·180 | 0·084 | 0·174 | 0·151 | 0·311 | 0·165 | 0·260 | 0·167 | 0·120 | 0·130 |
| Soluble ash | 0·192 | 0·200 | 0·311 | 0·194 | 0·090 | 0·042 | 0·128 | 0·051 | 0·224 | 0·136 | 0·128 | 0·084 | 0·045 | 0·053 |
| Insoluble ash | 0·070 | 0·040 | 0·049 | 0·058 | 0·090 | 0·042 | 0·046 | 0·100 | 0·087 | 0·029 | 0·132 | 0·080 | 0·075 | 0·077 |
| Potash | 0·106 | 0·110 | 0·210 | 0·119 | 0·051 | 0·024 | 0·081 | 0·034 | 0·151 | 0·078 | 0·088 | 0·047 | 0·045 | 0·044 |
| Sulphuric acid | 0·077 | 0·081 | 0·090 | 0·054 | 0·036 | 0·018 | 0·022 | 0·010 | 0·001 | — | 0·017 | 0·012 | 0·025 | 0·034 |
| Chlorine | 0·004 | 0·004 | 0·005 | 0·001 | 0·006 | 0·00 | 0·002 | 0·001 | 0·068 | 0·034 | 0·011 | 0·017 | — | — |
| Carbonic acid in soluble ash | 0·039 | 0·037 | 0·003 | 0·003 | — | 0·00 | 0·022 | 0·009 | 0·020 | 0·010 | 0·012 | 0·018 | 0·008 | — |

So far as we can judge from the limited data at our disposal, it is very probable that in ripe cane a high potash content accompanies a low quotient of purity in the juice and a high percentage of reducing sugar.

As regards the distribution of the mineral substances in a horizontal direction, we may quote an analysis of Bonâme, who found the rind of the cane to contain 0·61 per cent. of ash, against 0·25 per cent. in the pith.

It will be seen that the proportion in which the different constituents of our raw material occurs varies within rather wide limits. For ripe, sound, and normal cane, we may state the following limits, which will only be exceeded in certain rare cases :—

| | |
|---|---|
| Sucrose | 11-16 |
| Reducing Sugar | 0·4-1·5 |
| Fibre | 10-15 |
| Ash | 0·5-1·0 |

# PART II.

# SUGAR MANUFACTURE.

CHAPTER I.

# EXTRACTION OF THE JUICE.

## 1.—Mills.

The oldest and almost only method of extracting the juice is by means of mills. Formerly this was done on a small scale by crushing the canes between two wooden or stone cylinders, driven by hand or by bullocks, but now-a-days iron or steel rollers are employed, and driven by steam.

**Historical.**

Formerly the cane was crushed only once and the extracted juice boiled into sugar, but as the bagasse still contained a very considerable quantity of saccharine juice, which was lost, the present practice is to re-crush the bagasse from the first set of rollers between a second and a third or even a fourth set, so that multiple crushing is practiced everywhere. Moreover, some additional apparatus is employed for breaking or slicing the cane in order to relieve the work demanded of the rollers of the mill proper.

**Single and Multiple Crushing with Maceration.**

The most common form of mill is the three-roller type or combinations of the three-roller mills. Two of the rollers on the same level turn in the same direction and bear the names of "feed-roller" and "bagasse-roller" respectively, while the third, the "top-roller," is placed above the other two and turns in the opposite direction. As a rule the rollers of the first mill are placed in such a way that the cane enters easily and is but slightly squeezed, but the space between the top and the bagasse-rollers is so small that only the well crushed cane fibre can pass, and it is here that the actual crushing takes place. Care must, however, be taken that the forward motion of the crushed bagasse is not hampered by too small an outlet between the top and bagasse-rollers. The distance between the top-roller and the feed-roller and between the former and the bagasse-roller of the second mill is smaller than those of the first, and still smaller in the third one in order to obtain the maximum extraction.

**Three-Roller Mills.**

**Distance of the Rollers.**

The extracted juice falls from the rollers on to the bedplate of the mill, which has the form of a collecting trough with perforated bottom, which keeps back the

coarser particles of cane and allows the juice to pass, whence it is conveyed through open gutters to the liming tank.

In order to carry the canes or the bagasse from the feed-roller to the bagasse-roller, a curved metal plate, called the trash or bagasse turner, is fixed under the top-roller, but is capable of adjustment. The accurate adjustment of this plate is one of the principal points in mill work, and like the adjustment of the rollers, requires constant attention on the part of the engineer. It would be out of place to enter into details of the subject* in the present work, which is mainly addressed to chemists, but the following remarks may suffice.

**Bagasse Turner.**

The proper adjustment of the rollers, *i.e.*, the space between the feed-roller and the top-roller, and between the latter and the bagasse-roller, depends entirely on the amount of cane that has to be worked up in twenty-four hours with a given velocity of the mill, and on the quantity of juice that is allowed to remain in the bagasse, according to the capacity of the factory. The relation between those two spaces is not constant for different cane varieties but depends on the toughness of the fibre. The position and curve of the trash-turner exert a great influence on the efficiency of the mill work; but at present we do not know with sufficient accuracy how these should be modified to suit different circumstances. In practice, the tendency is to reduce the space between the bagasse-turner and top-roller as much as possible, without running the risk of jamming the cane fibre between them.

**Opening of the Mills.**

The space between the bagasse-turner and the bagasse-roller depends on the position of the mill in a combination of similar mills. It is larger in a first mill, where only canes are crushed, than in the second or third where the already finely divided bagasse has to be more heavily crushed.

**Speed of Rollers.**

No definite figures can be given as to the surface-speed of the rollers, but this should be as low as the required output will permit. The surface of the roller, whether rough or smooth, has also a great influence in this connection.

The exhausted bagasse passes out of the mill on the top of the bagasse-roller and gravitates down an inclined plate which is adjusted as near to the bagasse-roller as possible. It is here received upon an "intermediate" carrier which elevates and discharges it upon a second inclined plate whence it enters the second or third mill. The bagasse from the last mill falls on to a "bagasse-carrier," or into waggons or baskets by means of which it is transported to the furnaces.

**Intermediate and Bagasse-Carriers.**

Formerly the bagasse had to be sun-dried before it could be used as fuel, but in the last decade furnaces have been so improved that bagasse containing up to

* This subject is admirably treated by Noel Deerr on page 101 of his book.

50 per cent. of moisture may be used as fuel without drying it beforehand; the water content, however, must not exceed this figure, otherwise combustion is incomplete.

Mills with two rollers are also used, the rollers being placed one above another and the cane introduced between them by means of a carrier. Moreover we find mills having 4, 5, or 8 rollers, but, as already stated, the three-roller mill is the most common type. Mills with 9, 12 or 14 rollers are merely combinations of three-roller mills placed in tandem or combined with a preliminary "cane crusher" having only two rollers. These compound mills are placed very close together, and connected by means of intermediate carriers, the whole plant being driven by one or two engines by means of multiple gearing.

**Mills with 2, 4, 5, 8, 9, and more Rollers.**

Special appliances have been adopted in order to relieve the mills and enable them to grind more cane in a given time and with a given power, also with a view to disintegrating the whole cane, so that the bagasse passing out of the first mill may be sufficiently loose and finely divided to permit of its being thoroughly moistened by the maceration water which is applied at this stage. Such appliances include the Ross Cane Cutter, and the Cane Shredder. The former cuts the cane in pieces of about four inches and afterwards divides it into thin slices, while the latter reduces the cane to fibre between two rows of teeth before it enters the mill. If the knives of the Cutter and the teeth of the Shredder are sufficiently sharp, this can be effected without any loss of juice; the divided cane is afterwards crushed in the same way as the canes themselves, by mills, the rollers of which must be further apart than in the case of whole canes, because they receive them in a more voluminous form.

**Shredders and Crushers.**

A third type, the "Crusher," consists of a set of rollers provided with V-shaped teeth, which are placed so close together that the canes passing through them are slightly squeezed, and therefore the first mill can crush them more easily than it can whole cane. The Crusher extracts a part of the juice, and in that respect differs from the above.

In order to feed the mills regularly the canes are thrown on to long carriers, upon which they are spread out by hand before they reach the mill. In the case of fallen canes, which are usually crooked and therefore difficult to spread out, a spindle provided with revolving knives is sometimes adjusted across the carrier so that the canes passing under them are cut into short lengths and more evenly distributed across the carrier.

**Cane Carriers.**

When adopting double or triple crushing, it is advisable to use the most powerful mill as first mill, so that it may express 65 per cent. of the weight of the

**Power of the different Mills.**

cane, leaving only 25 per cent. of juice in the bagasse, when the fibre content is about 10 per cent. If higher, the first mill will not express so much. The following mills express a little more juice from the bagasse, but it is evident that even the heaviest pressure will fail to express all the juice. Owing to the colloidal water present in the fibre, the juice remaining in the bagasse will not have the same concentration as that which is expressed, but as it contains sugar some loss is inevitable.

**Maceration.**

When water is poured on the bagasse this residual juice is diluted, and after re-crushing the bagasse to its former content of juice, it will then contain the same amount of diluted, but therefore less saccharine, juice, causing less loss of sugar, so that maceration considerably improves the juice extraction.

Cane that has been crushed once, or in some factories even twice, and thus has lost a great deal of its juice, is macerated at the moment when the bagasse emerges from between the rollers, and (relieved from the heavy pressure) expands by its own elasticity and eagerly absorbs water. By the crushing in the mill the cell-walls have already been torn, allowing the maceration water at once to dilute the juice still remaining; additional water is poured on the partially exhausted bagasse on its way from one mill to another, and it is moreover turned again in some factories, causing the juice which is expressed by the next mill to be highly diluted. A calculation will make this point clear. Suppose that in a factory having triple crushing a maceration of 15 per cent. on the weight of cane is applied after the second mill on bagasse of cane that has already lost 72 per cent. of juice, and therefore still contains 18 per cent. of juice on the weight of cane, and that the third mill crushes this macerated bagasse again to the same content of juice as when it left the second mill, the following is the result, assuming that juice and maceration water have been completely mixed in the bagasse: The bagasse contained 18 per cent. of juice, water was added to an amount of 15 per cent.; therefore the macerated bagasse contained $18 + 15 = 33$ per cent. of diluted juice, of which $\frac{18}{33}$ is original undiluted juice. Now the third mill crushes the bagasse again to 18 per cent. of juice on the weight of cane, causing 15 per cent. of diluted juice, corresponding to $\frac{18}{33} \times 15 = 8{\cdot}2$ per cent. of original juice to be extracted, which, without maceration and recrushing, would have been lost.

It is true that in the short time that the bagasse requires to pass from one mill to another, the maceration water cannot mix completely with the juice still remaining in the bagasse; a mixture of 80 per cent. is very satisfactory.

In a microscopical inspection of macerated bagasse Went found that only one half of the cells were filled with liquid, while the other half had remained unmoistened, and, owing to this, the diluted juice extracted by the last mill does not contain so much sugar as is calculated here.

Supposing a mixture of 80 per cent., then 80 per cent. of the juice in bagasse or $\frac{18 \times 80}{100} = 14{\cdot}4$ parts of original juice mix with 15 parts of water. These 29·4 parts of diluted juice contain each $\frac{14{\cdot}4}{29{\cdot}4}$ parts of original cane juice; therefore, the 15 parts of juice extracted contain $\frac{14{\cdot}4}{29{\cdot}4} \times 15 = 7{\cdot}35$ parts of original juice, instead of the 8·2 per cent. which would be the case if the maceration water had completely mixed with the juice in the bagasse.

**Maceration with Last Mill Juice.**

In many sugar factories the bagasse is macerated between the first and second mills with the diluted juice from the last mill, and with pure water between the second and the last mill. All the juice coming from the last mill is poured over the bagasse passing from the first mill, and the juice from the first and second mill is conveyed into the clarifiers. This maceration with the last mill juice can only be applied if the cane is crushed so heavily in the first mill that the first bagasse can easily absorb liquid. A less powerful mill only squeezes the canes, and they leave it with a smooth surface and are unable to absorb the last mill juice. In this case this added saccharine liquid is again extracted by the second mill without having diluted the juice in the bagasse, or it soaks through the planks of the carrier on to the floor and is consequently lost.

**Condition of the Bagasse for Maceration with Last Mill Juice.**

By the use of such appliances as Cutters, Shredders, or Crushers, it is possible to obtain the first mill's bagasse in such a spongy condition that it eagerly absorbs the last mill juice and, therefore, after leaving the second mill, contains a more dilute juice than when the maceration with last mill juice has been omitted, or when that juice has only moistened its surface and has not penetrated into the interior of the layer of bagasse on the carrier. It is evident that the cane-preparing machines not only allow quicker working and decrease the risk of break-downs, but for the above reason also bring about a better juice extraction while maintaining the same dilution.

Müller von Czernicky * published the following data on the advantage of maceration with last mill juice:—

**Advantages of Maceration with Last Mill Juice.**

During 14 consecutive days cane was crushed by a set of three three-roller mills and macerated alternately one day with water and the next day with last mill juice. Great care was taken to grind about the same quantity of canes every day, also, that the canes were as similar as possible. The fact that he succeeded in both conditions is proved by the uniform composition of the first mill juice, and the constant fibre content of cane and bagasse.

* *Archief voor de Java Suikerindustrie*, 1899, 170.

| Maceration with Water. | | | | | | | | | Maceration with Last Mill Juice. | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Brix. Normal Juice. | Brix. Mixed Juice. | Maceration. | Brix. last Mill Juice. | Sucrose Bagasse. | Dry Substance Bagasse. | Fibre Bagasse. | Fibre Cane. | Sucrose lost on 100 cane. | Brix. Normal Juice. | Brix. Mixed Juice. | Maceration. | Brix. last Mill Juice. | Sucrose Bagasse. | Dry Substance Bagasse. | Fibre Bagasse. | Fibre Cane. | Sucrose lost on 100 Cane. |
| 17·7 | 15·7 | 12·5 | 7·16 | 4·07 | 55·0 | 48·0 | 11·05 | 0·97 | — | — | — | — | — | — | — | — | — |
| 17·3 | 15·2 | 14·0 | 6·54 | 4·03 | 54·7 | 47·5 | 10·9 | 0·92 | — | — | — | — | — | — | — | — | — |
| 17·5 | 15·2 | 15·5 | 6·35 | 3·90 | 55·8 | 47·3 | 11·2 | 0·92 | 17·4 | 15·3 | 13·5 | 5·69 | 3·77 | 55·9 | 47·4 | 11·2 | 0·89 |
| 17·3 | 15·3 | 12·7 | 6·50 | 3·97 | 56·4 | 46·6 | 10·7 | 0·91 | 17·5 | 15·7 | 11·0 | 6·38 | 3·88 | 56·1 | 48·3 | 11·1 | 0·89 |
| 17·7 | 15·5 | 13·9 | 6·51 | 4·09 | 55·8 | 47·5 | 11·1 | 0·95 | 17·6 | 15·8 | 11·3 | 6·40 | 4·00 | 55·8 | 48·4 | 11·2 | 0·92 |
| 17·6 | 15·6 | 12·7 | 6·49 | 4·02 | 56·9 | 47·8 | 10·8 | 0·91 | 17·6 | 15·9 | 10·7 | 6·52 | 4·03 | 56·1 | 47·6 | 11·0 | 0·93 |
| 17·5 | 15·2 | 15·2 | 6·37 | 4·03 | 56·1 | 47·6 | 10·7 | 0·91 | 17·3 | 15·4 | 12·4 | 6·37 | 4·10 | 56·4 | 47·8 | 11·0 | 0·94 |
| 17·6 | 15·3 | 15·2 | 6·40 | 4·08 | 54·5 | 47·2 | 11·5 | 0·99 | 17·4 | 15·4 | 13·1 | 6·27 | 3·95 | 55·5 | 48·1 | 11·1 | 0·91 |
| 17·7 | 14·9 | 18·7 | 6·32 | 4·05 | 55·0 | 47·4 | 11·0 | 0·94 | — | — | — | — | — | — | — | — | — |
| 17·5 | 15·3 | 14·5 | 6·51 | 4·02 | 55·6 | 47·4 | 11·0 | 0·94 | 17·5 | 15·6 | 12·0 | 6·27 | 3·76 | 55·7 | 47·6 | 11·0 | 0·91 |

We see that when macerating with last mill juice, 2·5 per cent. less water causes a smaller loss of sugar in bagasse than with water alone. The water so added is therefore used twice over, viz., once in the form of last mill juice of about 6·5 per cent. dry substance (in order to dilute the juice of 17·5 per cent.), and the second time in the form of water between the second and third mills to reduce the juice left in the second bagasse to the low density of the last mill juice.

**Practical Limits of Maceration.**

This process of continuous maceration and extraction, however, has its limits, as it is not economical to go on in this way in order to extract all the sugar from the cane, as in this case the cost of evaporating the huge quantities of maceration water would exceed the yield of the extra sugar thus obtained. Moreover, by the action of the repeated maceration and crushing, all kinds of bodies are extracted from the fibre and pass into the juice, causing it to become more impure and more troublesome to clarify.

Finally, as the fibre appears to absorb and retain the last traces of sucrose, the admixture of the juice in the bagasse with the maceration water becomes more and more incomplete in proportion as the amount of maceration water is increased. When much maceration water is added, the sucrose content of the bagasse does not diminish proportionally, and sometimes the sucrose content of the last expressed juice is actually inferior to that of the last bagasse, and accordingly much inferior to the juice present in it.

**Sucrose Content of Bagasse with different Amounts of Maceration Water.**

The following table gives the sucrose content of bagasse and of last mill juice in a great many Java sugar factories employing triple crushing and maceration with different amounts of maceration water applied to cane having very varying sucrose contents.

| Sucrose in Normal Juice. | Amount of Maceration Water in per cent. of Normal Juice. | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | Under 12. | | 12-14 | | 14-16 | | 16-20 | | Over 20. | |
| | Bagasse | Last Mill Juice. | Bagasse | Last Mill Juice. | Bagasse | Last Mill Juice. | Bagasse | Last Mill Juice. | Bagasse | Last Mill Juice. |
| 12/13 | 4·14 | 5·56 | 4·25 | 5·73 | 4·28 | 5·44 | 4·23 | 4·92 | — | — |
| 14 | 4·52 | 6·70 | 4·37 | 5·63 | 4·46 | 6·34 | 4·85 | 6·24 | 4·92 | 6·45 |
| 15 | 4·97 | 6·90 | 4·38 | 5·78 | 4·38 | 5·54 | 4·42 | 5·48 | — | — |
| 16/17 | 5·50 | 9·31 | 4·83 | 6·86 | 4·46 | 6·04 | 4·87 | 6·06 | 4·86 | 4·53 |

We see that, in Java, the maceration is practically limited to between 12 and 16 per cent., and that maceration with 20 per cent. does not produce such superior extractions as might be expected. The sucrose content of the bagasse from Java canes, of average composition, may be put down at 4·50 per cent., with small deviations above and below. According to figures from Hawaii, the extraction obtained with 12-roller mills is much better than this, but the quantity of maceration water added is also much more.

**Milling Results with a 12-Roller Mill.**

Hedemann reports the following data concerning the results of a 12-roller mill during a whole grinding season.

| | |
|---|---|
| Sucrose in cane | 15·37 |
| Fibre in cane | 10·98 |
| Sucrose in bagasse | 3·28 |
| Moisture in bagasse | 45·66 |
| Fibre in bagasse | 49·92 |
| Sucrose lost in bagasse on 100 cane | 0·76 |
| Extraction on 100 sucrose in cane | 95·40 |
| Dilution by maceration on 100 normal juice | 26·12 |

**Maceration with Hot or with Cold Water.**

There has been much difference of opinion as to the advantages of hot or cold maceration. Hot water has the disadvantage of dissolving more gummy matter from the bagasse and bringing it over into the juice, but still it is to be preferred from a practical point of view. The cold water used for the purpose in sugar mills is usually dirty and needs filtration, therefore with a view likewise of preventing the choking up of the apertures in the supply pipes or in the sprayers, it is better to use the condensed water from the hot well. As it is an advantage to force the water against the bagasse, the maceration supply is generally connected with the feed pump of the boilers. In this simple way one obtains a steady supply of hot water under considerable pressure.

Some believe that hot water mixes more readily with the juice in the bagasse than does cold, owing to a kind of diffusion which takes place between the juice in still living cells of the bagasse and the surrounding hot water, but which is

impossible in the case of cold water. But by microscopical inspection Went* showed that all of the cells are already broken by the heavy pressure of the first mill, so that, even if we admit a certain amount of diffusion through those ruptured cells, this takes place as easily with cold water as with hot.

Müller von Czernicky † carried out comparative experiments with hot and cold maceration and found no difference, as is seen in the following table :—

| | Cold Maceration. | Hot Maceration. |
|---|---|---|
| Brix first mill juice | 17·9 | 17·7 |
| Brix mixed juice | 15·9 | 15·9 |
| Brix last mill juice | 7·2 | 7·4 |
| Maceration on 100 parts of first mill juice | 11·3 | 11·0 |
| Sucrose in bagasse | 4·33 | 4·40 |
| Quotient of sucrose in bagasse and Brix last mill juice | 0·60 | 0·60 |
| Quotient of sucrose in bagasse and sucrose last mill juice | 0·78 | 0·78 |
| Quotient of purity first mill juice | 83·1 | 82·9 |
| Quotient of purity last mill juice | 76·7 | 76·5 |

Two further experiments showed that even after clarification no difference in the quotient of purity could be detected.

| | Cold Maceration. | Hot Maceration. |
|---|---|---|
| Increase in purity of the last mill juice by clarification | 3·7 | 3·8 |
| | 3·9 | 3·6 |
| Original purity | 74·8 | 74·4 |
| | 73·0 | 72·5 |

As the risk of dissolving gummy matter in hot water is not very great and as, on the other hand, no better extraction may be expected, it is immaterial whether hot or cold maceration be adopted, but from a practical standpoint the author would advise boiler-feed-water for this purpose.

**Method of Macerating with Last Mill Juice.**

Most factories pass the last mill juice through a strainer and apply it to the bagasse coming from the first mill without further treatment. In very rare cases this juice is clarified before being used for maceration. As such juice always contains cane fibre, it must be applied differently to the usual maceration water, which latter is forced against the bagasse through fine apertures or by means of Körting's injectors. In the case of last mill juice, it is preferable to employ an open, inclined gutter placed across the bagasse carrier.

**Maceration with Sweet Water.**

In factories which employ the carbonatation process the sweet waters from the presses (containing 1-2 per cent. of sucrose) are also employed for maceration and since they are free from fibre may also be supplied through pipes having fine apertures or by injectors.

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* *Archief voor de Java Suikerindustrie*, 1895, 673.
† *Archief voor de Java Suikerindustrie*, 1899, 174.

**Regulation of Crushing and Maceration.**

It is advisable in every factory to control the millwork according to the sucrose and water content of the last bagasse, and to regulate the maceration by the specific gravity of the last mill juice.

The more powerful the mills are, the more work may be claimed of them, and the drier and more exhausted will the bagasse be. But as extraction is not subject to fixed rules, no exact figures can be given. Factories equipped with a large evaporating plant and sufficient steam, may go farther with maceration than those where stoppage in the course of working, owing to want of capacity, is to be feared. Besides this, copious maceration will be more in place when crushing cane with juice of a high saccharine content, than in cases where poor cane has to be worked up. In any case the extent of the maceration is regulated by taking care to maintain the diluted juice from the last mill at the same specific gravity. When crushing canes having a rich juice, more maceration water will be required to dilute the last portions of it to the fixed specific gravity than when a cane with a poor juice is crushed, and thus the maceration is regulated by the constant testing of the last mill juice by the Brix hydrometer so as to obtain bagasse containing juice with an invariable quantum of dry substance. Owing to the circumstance that the Brix hydrometer only ascertains the quantity of dry substance and not that of sucrose, the sucrose content of the bagasse cannot be regulated in the same exact manner, but the proportion between dry substance and sucrose does not differ so much in the same factory that any great mistake can ensue.

**Influence of the Character of the Cane Fibre on the Extraction of the Juice.**

If it were possible to crush the bagasse of the different cane varieties always to the same juice content, it would be feasible to obtain a constant sucrose content in the bagasse by keeping the density of the last mill juice constant. The quantity of juice extracted from 100 parts of cane would then only depend on the amount of fibre, and this quantity diminishes for two reasons when the amount of fibre rises. Firstly, cane with much fibre contains a proportionately smaller amount of juice, and, secondly, the quantity of bagasse is increased, and hence also the quantity of juice which it contains and that of the sucrose in it which is to be eliminated.

It is not, however, quite so simple, because the fibre of every variety of cane has its own power to resist pressure. Thus the Java Seedling No. 247 yields a dry bagasse much more readily than No. 100 or the Black Java cane, and in most cases, canes having a high fibre content will yield bagasse, the fibre of which offers but little resistance to pressure. This to some extent compensates for the increased loss of sugar occasioned by the large amount of bagasse obtained from canes of high fibre-content.

From the average of a great number of determinations it is seen that a high fibre content of the cane corresponds with a high fibre content of the bagasse, so that a hard cane yields a drier and more exhausted bagasse than a soft one.

| Fibre in Cane. | 1904. | | 1905. | | 1906. | | Average. | |
|---|---|---|---|---|---|---|---|---|
| | Fibre in Bagasse. | Number of Cases. | Fibre in Bagasse. | Number of Cases. | Fibre in Bagasse. | Number of Cases. | Fibre in Bagasse. | Number of Cases. |
| 9·50—10·49 .. .. | 45·86 | 10 | 46·54 | 5 | 45·88 | 5 | 46·04 | 20 |
| 10·50—11·49 .. ... | 46·44 | 37 | 46·62 | 38 | 46·19 | 31 | 46·43 | 106 |
| 11·50—12·49 .. .. | 47·29 | 31 | 47·13 | 36 | 46·37 | 39 | 46·90 | 106 |
| 12·50—13·49 .. .... | 50·07 | 3 | 49·74 | 11 | 48·54 | 15 | 48·92 | 29 |
| 13·50 and above.. .. | — | — | 47·36 | 2 | 49·21 | 2 | 48·68 | 7 |

A few experiments published by Van Nouhuys* on milling results with different cane varieties are quoted here.

| Constituents. | Cheribon. | Striped Preanger. | No. 33*a*. | No. 36. | No. 139. | No. 247. |
|---|---|---|---|---|---|---|
| Sucrose first mill juice .. .. | 15·73 | 14·90 | 13·00 | 14·31 | 15·96 | 14·29 |
| ,, last mill juice .. ... | 8·98 | 7·90 | 7·68 | 7·39 | 11·13 | 7·39 |
| ,, mixed juice .. .. .. | 13·74 | 12·66 | 11·30 | 12·23 | 14·26 | 12·58 |
| Brix first mill juice.. .. .. | 17·4 | 16·9 | 15·6 | 16·5 | 18·6 | 16·5 |
| ,, last mill juice .. . .. | 10·6 | 9·4 | 9·8 | 9·1 | 13·8 | 9·0 |
| ,, mixed juice .. .. ... | 15·5 | 14·6 | 13·8 | 14·4 | 17·0 | 14·7 |
| Maceration, per cent. .. .. .. | 12·3 | 15·8 | 13·0 | 14·6 | 9·4 | 12·2 |
| Fibre in cane .. .. .. .... | 8·93 | 10·56 | 13·92 | 15·91 | 12·83 | 12·79 |
| Sucrose in bagasse .. .. .. | 5·89 | 5·90 | 4·15 | 3·28 | 5·87 | 4·01 |
| Moisture in bagasse .. .. ... | 48·63 | 51·51 | 40·95 | 36·86 | 47·55 | 42·09 |
| Fibre in bagasse .. .. .. . | 44·42 | 41·47 | 53·76 | 59·10 | 45·18 | 53·05 |
| Sucrose in cane .. .. .... | 13·51 | 12·43 | 10·53 | 11·12 | 12·85 | 10·22 |
| ,, lost in bagasse on 100 cane | 1·18 | 1·50 | 1·07 | 0·88 | 1·67 | 0·88 |
| ,, recovered in juice .... | 12·33 | 10·93 | 9·46 | 10·24 | 11·18 | 9·34 |
| ,, ,, on 100 sucrose in cane | 91·3 | 87·9 | 89·8 | 92·1 | 87·0 | 91·4 |

Very probably the considerable difference in juice content of bagasse of soft and hard canes, when crushed in the same mill, in a layer of equal thickness, is occasioned by the difference in absorbing power of the fibre. The fibre of soft cane is more spongy than that of hard cane, and therefore absorbs more juice than the latter. At the moment when the bagasse is relieved from the heaviest pressure, all of the expressed juice has not escaped out of contact with the bagasse and a film of juice still remains on the rollers, consequently the expanding fibre readily absorbs whatever juice remains in contact with it, although such juice has already been expressed from it. The more spongy the fibre the more it will absorb, and though this is still an unproved hypothesis it

* *Archief voor de Java Suikerindustrie*, 1904, 973.

may explain why soft cane yields bagasse containing so much more juice than does hard cane.

**Loss of Sucrose in Bagasse by Triple Crushing.**

The loss of sugar in bagasse on 100 cane has not been materially reduced in Java during the last few years, but the per cent. of sucrose in the bagasse has greatly decreased during that time. The milling plants have been improved, and at the same time the cultivation of canes carrying a higher percentage of fibre (and thus yielding a less resistant bagasse) has been extended, which two circumstances have tended towards reducing the sucrose content of the bagasse.

On the other hand, the high fibre content of the canes has increased the total amount of bagasse per 100 of cane, which causes the figures for sucrose lost on 100 cane to revert to their former value.

From data supplied by the Mutual Control of the Java Sugar Factories the figures for the extraction throughout Java in the last 10 years have been as follows:—

| Year. | Fibre in Cane. | Sucrose extracted on 100 Sugar in Cane. | Loss of Sucrose in Bagasse on 100 Cane. |
|---|---|---|---|
| 1899 | — | 90·3 | 1·36 |
| 1900 | 10·65 | 90·1 | 1·22 |
| 1901 | 10·95 | 90·2 | 1·24 |
| 1902 | 10·91 | 91·0 | 1·21 |
| 1903 | 11·21 | 90·6 | 1·17 |
| 1904 | 11·29 | 91·4 | 1·12 |
| 1905 | 11·61 | 90·4 | 1·12 |
| 1906 | 11·78 | 90·9 | 1·13 |
| 1907 | 12·00 | 91·2 | 1·15 |

## 2.—Diffusion.

Even with the best and most powerful mills, we do not succeed in expressing more than 90 to 96 per cent. of the sucrose of the cane. the result being that the balance of 10 to 4 per cent. is consumed in the fuel,* while at the same time the heavy pressure causes a great quantity of impurities to pass over into the juice. This, as well as the example of the beet sugar industry, induced the planters to try to substitute the diffusion process for the mill, but as yet this has not met with general approval, and has even turned out a complete failure in most cases.

The principle of diffusion is based on the property which many bodies possess, when in solution, of passing through a membrane, if on the other side

* In former years when the methods and mills were not so perfect as they are now, the amount of sucrose remaining in the bagasse was larger; losses of 12 per cent. and over of sucrose in cane were then no exception.

**Its Principle.** of it a liquid is present containing a smaller quantity of the like body in solution, this process going on until a state of equilibrium is attained between the liquids on each side of the membrane. The bodies having this property are principally those capable of becoming crystallized, so called crystalloids, while gummy and albuminoid matters, so-called colloids, diffuse very slowly or not at all. In the living cell the protoplasmic tegument of the cell wall cannot be regarded as a dialytic membrane, but as soon as the cell is killed by heat, or in some other way, the protoplasm becomes pervious.

Experiments by Went * showed that a temperature of 50° C. destroys the cells of sugar cane, so that at that temperature diffusion can take place.

In practical working, diffusion is carried out by slicing canes into chips by means of machines, and transferring the chips into large vessels called diffusers,

**Treatment of the Diffusion Battery.** where hot water is poured on them, causing the cells to die, and sucrose, glucose, acids and some incombustible matter to pass through the cell walls until the consistency of the fluid inside and outside of the cells has become the same. Afterwards the water, or more properly speaking the diffusion juice, is drawn off and poured upon a fresh quantity of slices, when it becomes still further enriched, and this process is repeated until the amount of sucrose in the diffusion juice has reached as nearly as possible that of the original cane juice. The slices that have been partially exhausted by the most concentrated juice have more dilute diffusion juice poured over them and finally water, and are totally exhausted, which operations, now that the cells are dead, may be affected without applying heat every time. Eventually, the slices, which have remained during all this time in the same diffuser, have successively been in contact with a diffusion juice of high concentration heated with steam to kill the cells, afterwards with more dilute juice and finally with water, so that when they are removed from the diffusion battery by opening the bottom of the diffuser they have a minimum content of sucrose.

The technical part, *i.e.*, construction and handling of the diffusion battery, may remain undescribed here, because it has already been frequently described in handbooks on the beet sugar industry. It may just be mentioned that the most advantageous way of heating is by steaming the diffuser filled with fresh chips to a temperature of 90° C., and not warming the others. At this temperature the cells die, and the sucrose diffuses, whilst albumen coagulates. After the diffusion juice is drawn off, its place is taken by juice which has been in contact with previously heated chips and hence has a lower temperature, and this goes on until the exhausted chips leave the diffusion battery at the same temperature as when entering it, so that no heat becomes lost in the chips. Nevertheless, experience has shown that with this mode of working almost all the sucrose of the cane is extracted, while at the comparatively low temperature prevailing in the battery, no inversion takes place and no organic substances are decomposed.

* *Archief voor de Java Suikerindustrie*, 1895, 672.

In the process of cane diffusion, reducing sugar diffuses the fastest, then sucrose, and afterwards some incombustible matters; albumen, colouring matter, pectine, &c., which cannot pass through the cell wall, are to be found in small quantity in the juice, having escaped from those cells which were ruptured in the slicing.

**Composition of the Juice in the different Vessels of a Diffusion Battery.**

The following table shows the composition of the juice in the different vessels of a diffusion battery:—

| No. of the Vessels. | Brix. | Sucrose. | Reducing Sugar. | Insoluble in Acidulated Alcohol. | Incombustible. | Not determined. | Quotient of Purity. | Glucose ratio. | Ash ratio. |
|---|---|---|---|---|---|---|---|---|---|
| Cane juice.. .... | 18·33 | 16·14 | 0·81 | 0·283 | 0·290 | 0·81 | 89 | 5·2 | 1·9 |
| 1.... .. .. .. | 12·6 | 10·74 | 0·58 | 0·117 | 0·170 | 1 | 85·2 | 5·4 | 1·6 |
| 2.. .. .. .. .. | 10·21 | 8·59 | 0·40 | 0·103 | 0·162 | 0·96 | 84·1 | 4·7 | 1·9 |
| 3.... .. .. .. | 7·1 | 5·95 | 0·25 | 0·085 | 0·135 | 0·68 | 83·8 | 4·2 | 2·3 |
| 4.. .. .. .. ... | 5·5 | 4·71 | 0·17 | 0·062 | 0·120 | 0·43 | 80 | 3·6 | 2·5 |
| 5.... .. .. .. | 4·4 | 3·71 | 0·15 | 0·067 | 0·109 | 0·36 | 84·3 | 4 | 2·7 |
| 6.. .. .. .. ... | 3·4 | 2·82 | 0·12 | 0·061 | 0·101 | 0·30 | 82·9 | 4·3 | 3·6 |
| 7.... .. .. .. | 2·81 | 2·26 | 0·08 | 0·058 | 0·085 | 0·33 | 80·6 | 3·6 | 3·8 |
| 8 . .. .. .. ... | 2·4 | 1·89 | 0·08 | 0·043 | 0·079 | 0·31 | 75·4 | 4·3 | 4·2 |
| 9.... .. .. .. | 1·9 | 1·42 | 0·04 | 0·030 | 0·081 | 0·33 | 74·7 | 3 | 5·7 |
| 10.. .. .. .. .... | 1·6 | 1·24 | — | 0·020 | 0·070 | 0·27 | 77·5 | — | 5·7 |
| 11.... .. .. .. | 1·2 | 0·99 | — | — | 0·054 | 0·16 | 82·5 | — | 5·5 |
| 12.. .. .. .. .... | 0·8 | 0·39 | — | — | 0·043 | 0·37 | 49 | — | 11 |
| 13.... .. .. .. | 0·5 | 0·28 | — | — | 0·031 | 0·19 | 56 | — | 11 |
| 14.. .. .. .. ... | 0·39 | 0·31 | — | — | 0·013 | 0·08 | 70 | — | 4 |

When, in the above list, the purity of the juice in No. 12 is found to be 49 and that in later ones higher again, we must bear in mind that the concentration of these juices is so small that a trifling and even insignificant dissolving of non-sugar from the fibre can considerably affect the figures for the purity. In the diffuser No. 12 a difference of 0·1° Brix is sufficient to change the purity by 7 degrees, so that we are not allowed to attach the same value to the purity of diluted solutions as to that of the higher concentrations we are accustomed to.

**Apparent low Purity at small Concentrations.**

Pellet* showed this clearly by dissolving 3 grammes of pure sucrose in water as is used for diffusion and ascertaining the purity of this solution in the same way as this figure is ascertained in the juice of the last diffusers. He found the figure 66 instead of 100, a warning for us not to attach too much value to the significance of quotients of purity of similar dilute solutions.

* *Bull. Assoc. Chim. Sucr. et Dist.*, 1896-7, 1070.

**The Working of a Diffusion Battery.**

With a view to obtaining some insight into the diffusion process the following experiments were made at Wonopringgo factory in Java. The battery consisted of 16 diffusers of 35 hectolitres capacity each, which contained 1735 kilogrammes of chips, and 17 hectolitres of juice were withdrawn each time from the last diffuser and pumped to the clarifiers, so that during the experiment, which began when the battery was in full swing, the work was conducted as regularly as possible. This appears also from the following table where all the figures for the content of the different diffusers for the bagasse and the waste water are brought together. After the exhausted chips had left the battery the superfluous water was expressed from them in a set of two mills. The sucrose content and the amount of the waste water were then ascertained and also the sucrose and fibre content of the bagasse, and the loss of sucrose calculated from these.

| Number of the Diffusers. | Sucrose in the Cane Chips. | Diffusion Juice. | | | Bagasse. | | | Waste Water. | |
|---|---|---|---|---|---|---|---|---|---|
| | | Brix. | Sucrose. | Quotient of Purity. | Grms. of Liquid in the Digestion of 40 grms. | Rotation in the 400 mm. tube. | Sucrose calculated from the former columns. | Rotation in the 400 mm. tube. | Sucrose. |
| 1 — 4 .. .. | 15·7 | 15·2 | 13·61 | 89·54 | 115 | 2·0 | 0·75 | 0·9 | 0·13 |
| 5 — 8 ... | 15·9 | 15·0 | 13·64 | 90·93 | 101 | 3·3 | 1·08 | 0·8 | 0·11 |
| 9 —12 .. .. | 15·4 | 15·4 | 13·77 | 89·42 | 102 | 2·6 | 0·87 | 0·6 | 0·08 |
| 13 —16 .... | 14·1 | 14·8 | 13·40 | 90·54 | 113 | 2·3 | 0·84 | 0·7 | 0·10 |
| $1^a$ — $4^a$ .. .. | 14·8 | 14·9 | 13·40 | 90·00 | 107 | 2·3 | 0·94 | 0·9 | 0·13 |
| $5^a$ — $8^a$ .... | 15·1 | 15·0 | 13·59 | 90·60 | 99 | 2·5 | 0·80 | 0·8 | 0·11 |
| $9^a$ —$12^a$ .. .. | 14·5 | 14·8 | 13·18 | 89·05 | 101 | 2·4 | 0·79 | 0·7 | 0·10 |
| $13^a$ —$16^a$ .... | 15·2 | 14·7 | 13·31 | 90·55 | 101 | 2·0 | 0·66 | 0·4 | 0·06 |
| $1^b$ — $4^b$ .. .. | 14·7 | 14·7 | 13·37 | 90·20 | 117 | 1·6 | 0·61 | 0·8 | 0·11 |
| $5^b$ — $8^b$ .... | 15·0 | 14·8 | 13·45 | 90·88 | 102 | 1·9 | 0·64 | 0·4 | 0·06 |
| $9^b$ —$12^b$ .. .. | 15·3 | 14·75 | 13·33 | 90·45 | 99 | 2·7 | 0·81 | 0·5 | 0·07 |
| $13^b$ —$15^b$ .... | 15·7 | 14·7 | 13·23 | 90·00 | 97 | 2·7 | 0·82 | 0·6 | 0·08 |
| Average .. . | 15·104 | 14·9 | 13·444 | 90·23 | — | — | 0·79 | — | 0·094 |

Fibre in cane .. .. .. .. .. .. .. .. .. .. .. 10·901 per cent.

Fibre in bagasse.. .. .. .. .. .. .. .. .. .... 9·947 ,,

Sucrose in bagasse on 100 cane $\frac{10{\cdot}901 \times 0{\cdot}79}{9{\cdot}947}$ .. .. 0·86 ,,

Sucrose in crushed bagasse .. .. .. .. .. .... 1·30 ,,

Moisture in crushed bagasse .. .. .. .. .. .. 61·74 ,,

Specific gravity diffusion juice.. .. .. .. .. .... 1·0609 ,,

| | | | |
|---|---|---|---|
| Weight of cane .. .. .. .. .. .. .. .. .. .. | | 81755 | kgs. |
| Weight of waste water .. .. .. .. .. .. .. .... | | 79900 | ,, |
| Loss of sucrose in waste water on 100 cane $\frac{0{\cdot}094 \times 79900}{81755} =$ | | 0·092 | per cent. |
| 81755 kgs. cane @ 15·105 per cent. sucrose = 12349 kgs. or | | 15·105 | ,, |
| 799 hectolitres juice @ 1·0609 = 847659 kgs. @ 13·444 .. .. .. .. .. .. .. .. | 11398 kgs. or | 13·905 | ,, |
| Total loss on 100 cane .. .. .. .... | 951 kgs. | 1·200 | ,, |
| Loss of sucrose in bagasse on 100 cane.. | 0·875 | | |
| And in waste water .. .. .. .... | 0·092 | | |
| Total of losses accounted for .. .. .. .. .. .. | | 0·967 | ,, |
| Unaccountable loss during diffusion .. .. .. .... | | 0·233 | ,, |

The known losses are therefore 0·97 per cent. on cane, and there is also an unexplained loss of 0·23 per cent., but this is perhaps fictitious, since it includes all the errors of the measuring of the juice, the weighing of the canes, and the sampling and analysis.

**Unknown Loss during Diffusion.**

This unexplained loss has been a source of much controversy in the literature of the beetroot sugar industry. Some investigators found considerable loss, others none at all, and the final conclusion was that these losses are inconsiderable, but that the analytical errors are sufficient to allow of a difference of 0·3 per cent. sucrose in the chips.* As the above figure falls within these limits, we may disregard the unexplained loss and only consider the losses actually accounted for, which amount to 0·97 per cent. or 6·4 per cent. on 100 sucrose in cane, which indicates an extraction of 93·6 per cent.

**Loss of Sucrose on Diffusion.**

The losses of sucrose during the grinding season at two Java factories were as follows :—

| Estate. | Sucrose in Cane. | Sucrose extracted in juice on 100 Cane. | Sucrose extracted on 100 Sucrose in Cane. | Sucrose lost. | | Dilution on 100 Juice. |
|---|---|---|---|---|---|---|
| | | | | On 100 Bagasse. | On 100 Sucrose in Cane. | |
| Wonopringgo Estate, 1899 .. | 14·95 | 14·21 | 95·00 | 0·74 | 5·00 | 28·6 |
| ,, ,, 1900 .. | 14·08 | 13·35 | 94·82 | 0·73 | 5·18 | 30·3 |
| Klaling Estate, 1901 .. | 11·97 | 11·34 | 94·74 | 0·63 | 4·26 | 25·9 |

Diffusion thus extracts 95 per cent. with over 25 per cent. dilution, or only 2 per cent. more than triple crushing with 14 per cent. dilution.

* *Sucrerie Belge*, 15th May, 1905.

The contest between diffusion and milling is decidedly in favour of the latter process, due to the many improvements made in mill and supplementary appliances in the last few years. The extraction by means of mills is no longer so inferior to diffusion work as it used to be. Further, the construction of the mills has also been much improved, so that breakdowns occur less frequently and repairs are more easily affected than in former times. Finally, the introduction of Cutters and Shredders has contributed considerably to a more regular working of the whole milling plant.

**Contest between Diffusion and Mills.**

One great advantage of mills is that they permit of more variations in output than is the case with the diffusion battery. When, for instance, burnt cane must be crushed at once, it is possible to crush 600 tons a day with a milling plant intended to crush, say 450 tons, provided the other departments of the factory can handle the additional juice. It is evident that such an increase of work cannot but influence its quality, so that a poorer extraction will be the consequence; and granting this, we must not overlook the fact that a diffusion battery may also accomplish such additional work. But allowing for a very poor extraction, it would not be feasible to work up all the canes by diffusion, as the slicing machine might be unable to deal with the surplus cane. On the other hand, the advantages of diffusion are that it extracts nearly all the sugar from the cane in the form of a pure juice, the greater part of the impurities remaining behind in the bagasse, so that the juice is easily worked. There is also less danger of stoppages, for if one diffuser is out of order, it is disconnected and the work carried on with the others.

**Advantages of the Milling Work.**

The disadvantages are that more hands are required for the diffusion system, and that they must be chiefly workmen specially acquainted with the routine. Moreover, the diffusion juice is very much diluted (up to 20 per cent. and sometimes beyond), and a large quantity of steam is required to evaporate the diluting water. It is true that in some factories, working with mills, the dilution by maceration is not less than 20 per cent., but only when crushing canes with a high saccharine content which renders such dilution worth while; on the other hand, as it is not possible in diffusion to diminish the dilution in the case of poor cane, poor and rich juices are equally diluted.

**Disadvantages of Diffusion.**

Finally, the slicing machine consumes a great deal of steam, and, as it has very heavy work to do, constant repairs are necessary. The exhausted slices contain so much water that they have to undergo preliminary crushing in a mill before being dried in the sun to form serviceable fuel. Naturally the mill has less work to do than when it is used for cane crushing, but still it is a great inconvenience to be obliged to have a mill along with the diffusion battery. Then the bagasse is much finer and more powdery than mill-bagasse, and is therefore less valuable as fuel. Against the advantages of extracting more sugar and having a purer juice, and more regular working, we have the disadvantages of requiring more and also higher skilled workmen, being compelled to use more

steam, and getting a less valuable fuel as an after-product, whilst the capacity of the battery does not adapt itself to a decrease or increase in the quantity of cane to be worked up as does a milling plant.

Finally, at every stoppage of the diffusion battery it is necessary either to complete the "round" of the battery at the cost of a heavy dilution, or to discharge the chips when only partially exhausted.

**Loss of Time and Sugar at Stoppages.**

Ordinarily a compromise is made between these alternatives, necessitating a considerable loss of saccharine matter as well as a great dilution which costs much valuable fuel. As the cane is not stored, and the supply may be very irregular in rainy weather, this drawback connected with the stopping of a diffusion battery is of a much more serious character than in Europe.

In the case of milling, stoppages do not entail such inconveniences.

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## 3.—Lixiviation of the Bagasse.

Within the last few years endeavours have repeatedly been made to combine milling with diffusion, the greater part of the juice being expressed by the mill,

**Principle.**

and the resulting bagasse submitted to diffusion or lixiviation in order to extract the residual juice. As such a combination would avoid the chief objections to diffusion it would have many advantages if only it could be applied in a practical manner.

As the mill breaks open and tears up the cells, no slicing machine is required, and the bagasse from the first or second mill is suitable for lixiviating. Further, 68 per cent. on cane or 80 per cent. of the total juice is obtained in an undiluted form and only 20 per cent. remains in the bagasse. It is evident that in such a case a large dilution of a small quantity of juice permits of extracting all the sugar, without the dilution (as calculated on 100 normal juice) becoming any higher than in mill work.

Such a method has been applied in Egypt by Perrichon.* The bagasse from single or double crushing is taken from the mill by a carrier and loaded into

**System of Perrichon.**

specially constructed waggons—a sort of tank on wheels, fitted with a strainer extending over the whole of the bottom, and a discharge valve; each contains 90 hectolitres of bagasse. One hectolitre of compressed bagasse weighs 20 kilogrammes, the total weight of the contents of each waggon will, therefore, be about $1\frac{3}{4}$ tons. Hot water is next run on to the bagasse from a service tank, and allowed to remain for about seven minutes, after which it is let off into a tank placed under the waggon. These washings are then pumped up into another tank, placed on the same level and in a line with

* *Journal des Fabricants de Sucre*, 1900, No. 41. *Sugar Cane*, 1898, 242.

the service tank, again heated to the desired temperature and allowed to flow on to another waggon load of bagasse ; after seven minutes this washing water is in turn drawn off, pumped up into another tank, and used afresh in the same manner. This operation is repeated nine times, the washings passing from tank to tank in the direction of the boiler house. The same water is thus passed through nine different waggon loads of bagasse and when it leaves the ninth waggon what was originally five tons of water has become only one-third of that quantity, having a density of 4° Beaumé. It is now a fine clear liquor, and is sent forthwith to the clarifiers, while the washed bagasse is passed through a mill to remove the remaining water before being dried for fuel.

In 1899 this process was applied in the Egyptian sugar factory "Rodah" during 96 days, crushing 1200 tons of cane per 24 hours. The cane was crushed in two mills without maceration and the bagasse lixiviated in two batteries of eleven tanks each. During that period the sucrose content of the cane was 12·2 per cent., the loss of sucrose in bagasse 0·4 per cent. on 100 cane or 3·3 per cent. on 100 sucrose in cane, which is equivalent to an extraction of 96·7 on 100 juice in cane. The Brix of the normal juice was 15·5, that of the lixiviated juice 10·7, giving a dilution on 100 parts of the juice of 45·3 per cent. The greater part of the juice, however, was extracted undiluted, which decreases the dilution calculated to the whole amount of juice. 100 kilogrammes of cane yielded 89·53 litres of total mixed juice having a specific gravity of 1·04 and, therefore, weighing 94·9 kilogrammes. These data do not allow the exact calculation of the dilution, but starting from the figures for the relation between weight of cane and of juice on triple crushing and maceration with known quantities of water we may take the dilution as about 15-16 per cent.

If the sucrose content of the water expressed on recrushing is taken into account, we find here a very good extraction of 96·7 on 100 juice, but the reports do not mention the fact that the bagasse could be squeezed dry enough to be burnt in the furnaces without artificial drying, which, of course, is almost as important a factor as the low sucrose content of the bagasse.

**Geerligs-Hamakers System.**

In the year 1903 a number of experiments were made at Wonopringgo estate in Java* with lixiviation of the bagasse in the same diffusion battery as mentioned on page 110.

The experiments were made in a diffusion battery, which was originally used for the diffusion of cane-chips, and had a capacity of 250-300 tons of cane per 24 hours. The capacity of the battery proved sufficient for the maceration of the bagasse from 450 tons of cane per 24 hours, with a loss of 0·25 parts of sucrose on 100 parts of cane and a dilution of 19·6 parts on 100 parts of normal juice. As the amount of cane crushed was about 2000 tons, these experiments represent regular factory work and not merely laboratory experiments, they showed further that for effective lixiviation the bagasse should be fine and regularly crushed,

* *International Sugar Journal*, 1903, 571.

such as can be obtained by means of a Cane Shredder or Crusher and double crushing. The increased extraction brings about solution of non-saccharine matter, so that where the purity of the juice extracted from the bagasse by a third mill was six points below that of the first mill juice, the juice extracted from that bagasse during lixiviation had a quotient of purity from eight to eleven points below that of the same first mill or normal juice. The clarified juice from the mixture of mill juice and lixiviation juice was 0·7 degress less in purity than the clarified juice from the same cane extracted by triple crushing. The moist bagasse from the maceration vessels could easily be deprived of its superfluous water by a three-roller mill, and then contained from 45-50 per cent. with an average of 48 per cent. water; it could be burned very satisfactorily in the furnaces without preliminary drying in the sun.

An extraction of 98 per cent. on 100 juice was thus obtained with a dilution of 19·6 on 100 normal juice and a slight decrease in purity of the juice as compared with milling. Apart from the reduced purity of the clarified juice, no difficulties were experienced with the process, and exhausted molasses (having a quotient of 30) were as easily obtained from this juice as from that extracted by mills. Only the second masse cuites were more viscous than usual.

The chief difficulty was in transporting the wet exhausted bagasse. This heavy and troublesome material gave much more trouble in the diffusion battery than the sliced cane-chips for which it was originally designed, and it was obvious that the diffusion vessels were not of the most suitable form for filling with and discharging the bagasse, and the circulation of the juices was much retarded.

In order to repeat the experiments on a proper basis, a new diffusion battery was necessary, but as the owners of the factory had already discarded the diffusion plant and were well satisfied with their new triple crushing plant, they were unwilling to spend money in new apparatus, the more so because no patent law exists in Java and the profit of their expenses would very probably be reaped by others who would at once copy the machinery as soon as it had proved a success. So this system has never gone further than the experimental stage with inadequate machinery.

In some factories in the Antilles, and in Madeira, Naudet's diffusion process of the bagasse is in use, but it does not seem to extend its application. Properly speaking it is just like ordinary diffusion but with forced circulation of the juice in the first vessel, as the juice is pumped several times through the bagasse contained in it, while at the same time heating it to a high temperature.

The cane is crushed by a crusher and two mills without maceration, and the resulting bagasse conveyed to a vessel which, for convenience' sake, may be termed a "diffuser." The cells in the bagasse having been torn asunder and thoroughly opened up by the shredder and mill, their contents are free to mix with the juices and water subsequently brought into contact with them. When the diffuser is

almost full of bagasse, cold mill juice is introduced which has the effect of compressing the loose bagasse and causing same to occupy a smaller volume, so that further additions of bagasse can be made until the diffuser is quite full. We now find that the constituents of the cane which were separated in the mill are here brought together in the diffuser and in the proportions in which they originally existed in the cane. The quantities of juice and bagasse which enter into the diffuser may amount to 58-60 kilogrammes per hectolitre capacity, while the balance of about 42-45 litres per hectolitre is supplemented by juice from a receiving tank, and as this tank is kept filled with juice drawn from the second diffuser, it contains the most concentrated juice of the whole battery. The first diffuser thus contains per hectolitre capacity 58-60 kilogrammes of cane, with its undiluted juice, and 45-42 litres of juice of maximum density. The bottom of the first diffuser is then put into communication with a mixing trough containing a measured quantity of juice, and which in turn is connected with a centrifugal pump. The juice is drawn from the said diffuser by this pump, passed through a juice heater (which rapidly heats it to its boiling point) and returned to the top of the same diffuser. During this circulation the juice also passes into the mixing tank, where it is limed and mixes with the juice already there. On passing through the heater, it becomes defecated and returns to the top of the diffuser with a temperature slightly above boiling point. During its downward passage through the closely packed bagasse, the juice parts with its precipitated impurities, and finally leaves the bottom of the diffuser in a defecated and filtered condition. Circulation is stopped as soon as the juice coming from the diffuser has a temperature of about 220°, this being reached after five or six minutes. Thereupon the juice is forced out of the diffuser (by means of compressed air or by water) into a tank, which supplies the evaporators, the larger portion of clear defecated juice being at once evaporated and the smaller part being used for wetting the bagasse in the next diffuser in the manner already described, this new diffuser having now received its charge of 56-60 kilogrammes of cane per hectolitre. The further working of the battery is similar to ordinary diffusion. The bagasse saturated with the concentrated juice is successively washed by the dilute juices, passing from diffuser to diffuser, and this goes on until each diffuser, in turn, becomes the last when it is filled with pure water which extracts the last traces of the juice. As the bagasse is finely divided and hot, the admixture of the juice in the bagasse and the surrounding water goes on very rapidly, so that the extraction is even more rapid than in ordinary diffusion since, in the latter, an appreciable time is necessary for the crystalloids to permeate the cell walls of the sliced cane. The bagasse is sterilized by the hot juice, thus preventing any fermentation or sourness.

The Naudet process is, therefore, not only designed to extract the juice from the cane but also to defecate and filter it. When making refining crystals the juice from the diffusers may be pumped without further treatment to the

evaporators, and is even then so white and pure that it is difficult to make it into brown sugar of the desired colour and polarization. For yellow and white sugars the raw juice still requires sulphitation, and the defecated juice an additional filtration through any mechanical filter, but this presents certain difficulties to be mentioned subsequently. The exhausted bagasse is passed through a double-crushing plant and used as fuel.

In this process the subsiding, decanting, and filtration of the "bottoms" is avoided, as also is the loss of sucrose in filter press mud, because the precipitated impurities which adhere to the bagasse are submitted to the same number of washings as the latter—first with juices of gradually diminishing densities, and finally with water.

The fact that the hot limed juice dissolves gummy matters from the bagasse is certainly a disadvantage, but not a very serious one. The owners of a factory where the Naudet process is in use state that even fourth sugar could be cured in the centrifugals without adding water, so that the increased viscosity of the molasses (due to gummy matters in the juice) was not sufficient to prevent their separation from the sugar crystals during curing.

Mr. Robert Harvey, whose firm constructed the Naudet machinery for "Caroni" Estate, Trinidad,* wrote an article on the subject, from which I quote the following particulars:

"In the year 1904 an extraction of 95 per cent. of the sucrose in the cane was obtained by this process at Messrs. Hinton's factory in Madeira. At Fortuna estate in Porto Rico the cane was crushed by one crusher and one mill, and the resulting bagasse treated by Naudet's process; 96 per cent. of the sucrose in the cane was recovered in the juice with a total dilution of 9 per cent. on 100 cane (equal to about 10 per cent. on 100 juice).

"The juice from the battery had a density of only 1·3° Brix below that of the original juice extracted by dry milling, and also the same quotient of purity."

The figures communicated to the writer by the same authority regarding the results obtained in 1906 at Caroni, in Trinidad, are not so good, but from his own experience of a similar process tried at Wonopringgo estate in Java, they appear to be quite satisfactory. In 1906 about 48,000 tons of cane were crushed at Caroni by a shredder and one mill, which extracted 62·4 per cent. juice from 100 cane. The resulting bagasse was extracted in a battery of 10 diffusers, of such capacity that each contained the bagasse yielded by three tons of cane. On an average, 25 tons of cane were worked per hour, thus bringing the interval between the filling of two subsequent diffusers to seven minutes. From every diffuser 30 hectolitres of juice were drawn off and sent direct to the evaporators. The exhausted bagasse was re-crushed in a mill, but as it was very fine and tended to choke the dumb-returner, the mill had to be opened to such an extent

* *International Sugar Journal*, 1905, 326.

that it was impossible to obtain bagasse containing less than 57·5 per cent. of water. The sucrose in the dry bagasse amounted to 1·80 per cent., and in the expressed water to 0·98 per cent.

The average figures for the extraction during the whole season are as follows :—

| | On 100 Cane. | On 100 Sucrose in Cane. |
|---|---|---|
| Sucrose in cane | 12·86 | 100· |
| Sucrose in juice | 11·93 | 92·8 |
| Sucrose lost in extraction | 0·93 | 7·2 |

The composition of the juices was :—

| | Mill Juice. | Mixed Juice. |
|---|---|---|
| Brix | 17·70 | 13·90 |
| Sucrose | 15·16 | 11·71 |
| Quotient of purity | 85·7 | 84·2 |

Dilution on 100 parts of normal juice, 27·3

So far, we see that lixiviation of the bagasse does not furnish a better extraction than triple crushing with maceration, and that a high dilution of the juice is an essential feature. The moisture-content of the bagasse was also very high, so that additional fuel was necessary in all these experiments.

The improvements made in the Naudet process subsequent to 1906 are not considerable, and from the author's own experience with the process in the Antilles it is very doubtful whether the advantage of eliminating the open clarification, re-subsiding and filter-pressing, together with the loss of sucrose in filter press mud, is great enough to compensate for the great amount of trouble in handling and drying the bagasse and the risk of losing sucrose in juice which after being extracted by the mill is poured back on the bagasse again and washed out in the battery.

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## 4. Composition of the Juice.

**Difference between Juice extracted by Mills and by Diffusion.**

As will follow from the observations made in the preceding chapter, the composition of cane-juice as expressed by the mill is quite different from that obtained by diffusion, the latter containing considerably less impurities.

Cane juice, as it comes from the mills, is an opaque liquid, covered with froth, owing to the imprisoned air bubbles it contains, and having a coloration varying from grey to dark green according to the colour of the cane from which it has been expressed. It contains in solution all the soluble constituents of the cane—sucrose, reducing sugar, salts, organic acids, and pectin, and besides the

air already mentioned it carries in suspension fine bagasse fibre, wax, sand and clay (adhering to the cane), colouring matter, and albumen, which latter body occurs in considerable quantity whenever unripe canes or green cane tops have been crushed with the ripe canes.

Fresh mill juice has an acid reaction, and, owing to the presence of albumen and especially pectin, is a viscous liquid that cannot be filtered cold, hence the suspended constituents cannot be removed without steaming, and we have therefore to be content with keeping back the larger particles of bagasse by means of copper strainers.

**Proportion in which the Constituents occur in the Juice.**

We have already seen from the data given in Chapter II., Part I., that the proportion in which the chief constituents occur in the juice varies greatly, and depends, among other things, on the variety and age of the cane, the nature of the soil, the manures applied, the climate, weather, &c. In the main, the figures for sucrose and reducing sugar show the greatest differences, while the other elements do not vary much, at least in sound and ripe canes. In unripe cane the juice sometimes contains much dissolved albumen, which forms a thick layer of froth in the measuring tanks, and as this froth remains on the top of the liquid for a long time it hinders the measuring of the juice. Later on, at the clarification stage, this albumen coagulates and is easily removed in the scums. A quantity of this froth was removed from the measuring vessels and gutters, washed, dried, and analysed, with the following results:—

| | |
|---|---|
| Cane Fibre | 62·50 |
| Wax | 4·12 |
| Albumen | 26·25 |
| Moisture | 7·13 |
| | 100· |

In dead or burnt cane the amount of free acid is larger than ordinarily occurs, but on the whole the other constituents do not show any considerable deviations.

Apart from the difference between the juice of different kinds of cane, the same cane may yield juice of a varying composition, according to the pressure it has undergone in the mill.

**Influence of Mill Power on the Proportion of the different Constituents of the Juice.**

This phenomenon is easily explained when one remembers how many tissues having different degrees of hardness and containing different kinds of sap are present in the cane.

The sap in the vacuoles contains nothing but sucrose, reducing sugar, a little salt, and organic acids. The protoplasm consists for the most part of water,

**Different Kinds of Sap in the Cane.**

which contains albumen and salts; and finally the sap in the fibro-vascular bundles also holds salts in solution but no sugar. We find therefore not less than three different kinds of sap in the cane, viz., that of the vacuoles, of the protoplasm, and of the fibro-vascular bundles. In the former and the latter the constituents occur only in solution, whereas that of the protoplasm contains suspended matters also. The softest tissues contain the purest and richest sap, so that this is expressed by the gentlest pressure. With a heavier pressure this juice is mixed with that of the protoplasm, whilst the fibro-vascular bundles part with their watery juice only under the heaviest pressure. Hot maceration causes the soft parenchyma cells to swell, and during re-crushing a quantity of finely divided cellular matter passes into the juice.

Moreover the hard, woody rind is only broken by the heaviest pressure, hence the second and third mill juices are much darker in colour than the first. The only thing that seems improbable is that the first mill juice is richer in glucose than the later ones, but this is ascribed to the top of the cane being the softest part and also containing the most glucose. Gentle pressure therefore expresses this easily and imparts to the first juice proportionally more glucose than to the other juices, which are mainly derived from the older tissues of the cane.

**Composition of Mill Juices.**

The following analyses illustrate the differences in the juices obtained by three successive crushings of the same canes with and without maceration.

I.—Triple Crushing, without Maceration.

*Black Java Cane.*

| Constituents. | First Mill. | Second Mill. | Third Mill. |
|---|---|---|---|
| Brix | 19·2 | 19·3 | 19·0 |
| Sucrose | 16·49 | 16·33 | 15·95 |
| Reducing sugar | 1·98 | 1·57 | 1·52 |
| Ash | 0·28 | 0·41 | 0·42 |
| Gum and pectine | 0·125 | 0·376 | 1·250 |
| Albumen | 0·025 | 0·092 | 0·054 |
| Free acid | 0·048 | 0·072 | 0·096 |
| Quotient of purity | 85·9 | 84·4 | 84·0 |
| Glucose ratio | 12·1 | 9·6 | 9·5 |
| Coloration | light. | dark | very dark |

## II.—Triple Crushing, with Maceration Water added after the Second, and Last Mill Juice added after the First Mill.

*Borneo Cane.*

| Constituents. | First Mill. | Second Mill. | | Third Mill. | |
|---|---|---|---|---|---|
| | | Diluted Juice. | Calculated on Brix=18·5 | Diluted Juice. | Calculated on Brix=18·5 |
| Brix | 18·5 | 14·8 | 18·5 | 6·7 | 18·5 |
| Polarization | 16·76 | 12·65 | 15·81 | 5·50 | 15·18 |
| Purity | 90·59 | 85·47 | 85·47 | 82·10 | 82·10 |
| Reducing sugar | 0·89 | 0·59 | 0·74 | 0·33 | 0·90 |
| Glucose ratio | 5·3 | 4·7 | 4·7 | 6·0 | 6·0 |
| Ash | 0·244 | 0·425 | 0·531 | 0·199 | 0·550 |
| Free acid as $SO_3$ | 0·042 | 0·039 | 0·048 | 0·016 | 0·044 |
| Combined organic acid as $SO_3$ | 0·077 | 0·080 | 0·100 | 0·027 | 0·073 |
| Albuminoids | 0·024 | 0·068 | 0·085 | 0·047 | 0·138 |
| Gums | 0·063 | 0·150 | 0·185 | 0·099 | 0·273 |

## III.—Triple Crushing, without Maceration.*

*Purple Cane. Second Year Ratoons.*

| | Per cent. |
|---|---|
| Extraction First Mill | 64·50 |
| ,, Second Mill | 5·50 |
| ,, Third Mill | 2·13 |
| Total Extraction | 72·13 |

| | First Mill. | Second Mill. | Third Mill. |
|---|---|---|---|
| Brix | 15·36 | 14·60 | 14·60 |
| Sucrose | 12·93 | 11·41 | 11·30 |
| Reducing sugar | 1·54 | 1·29 | 1·23 |
| Ash | 0·37 | 0·58 | 0·77 |
| Albuminoids | 0·18 | 0·50 | 0·58 |
| Free acid | 0·10 | 0·11 | 0·14 |
| Combined acid | 0·14 | 0·15 | 0·12 |
| Gums | 0·10 | 0·56 | 0·51 |
| Coefficient of purity | 84·07 | 78·15 | 77·39 |
| Glucose ratio | 11·91 | 11·30 | 10·88 |

* *Bulletin No. 91, Louisiana Exp. Station*, 31.

## IV.—Triple Crushing, with Steam Maceration.*

*Purple Cane.—First Year's Ratoon.*

| | Per cent. |
|---|---|
| Extraction, First Mill .. .. .. .. .. .. .. | 64·31 |
| ,, Second Mill .... .. .. .. .. .... | 8·00 |
| ,, Third Mill .. .. .. .. .. .. .. | 4·52 |
| Total Extraction .. .. .. .... | 76·83 |

| Constituents. | First Mill. | Second Mill. | | Third Mill. | |
|---|---|---|---|---|---|
| | | Diluted Juice. | Calculated to Brix = 15·84. | Diluted Juice. | Calculated to Brix = 15·84. |
| Brix .. .. .. .. | 15·84 | 12·05 | 15·84 | 11·53 | 15·84 |
| Sucrose .. .. ... | 13·50 | 9·40 | 12·31 | 8·70 | 12·01 |
| Reducing sugar.. .. | 1·37 | 0·97 | 1·27 | 0·88 | 1·21 |
| Ash .. .. .. ... | 0·37 | 0·57 | 0·75 | 0·65 | 0·90 |
| Albumen .. .. .. | 0·14 | 0·28 | 0·37 | 0·20 | 0·28 |
| Free acid .. ... | 0·09 | 0·13 | 0·17 | 0·14 | 0·19 |
| Combined acid .. .. | 0·13 | 0·16 | 0·21 | 0·19 | 0·27 |
| Gums .. .. ... | 0·24 | 0·54 | 0·70 | 0·83 | 1·14 |
| Purity.. .. .. .. | 85·23 | 78·00 | 78·00 | 75·45 | 75·45 |
| Glucose ratio.. .... | 10·01 | 10·32 | 10·32 | 9·42 | 9·42 |

## V.—Triple Crushing, with Cold Maceration 20%.†

*Purple Cane.*

| | Per cent. |
|---|---|
| Extraction First Mill .. .. .. .. .. .. .. | 66·50 |
| ,, Second Mill .. .. .. .. .. .... | 12·15 |
| ,, Third Mill .. .. .. .. .. .. .. | 4·51 |
| Total extraction.. .. .. .... | 83·16 |

* *Bulletin No.* 91, *Louisiana Exp. Station*, 31.

† *Bulletin No.* 91, *Louisiana Exp. Station*, 32.

| Constituents. | First Mill. | Second Mill. | | Third Mill. | |
|---|---|---|---|---|---|
| | | Diluted Juice. | Calculated to Brix = 15·73. | Diluted Juice. | Calculated to Brix = 15·73. |
| Brix .. .. .. .. | 15·73 | 10·48 | 15·73 | 6·93 | 15·73 |
| Sucrose .. .. ... | 14·01 | 8·65 | 12·97 | 5·64 | 12·80 |
| Reducing sugar.. . | 0·83 | 0·46 | 0·69 | 0·34 | 0·78 |
| Ash .. .. .. .... | 0·35 | 0·37 | 0·55 | 0·31 | 0·71 |
| Albumen .. .. .. | 0·12 | 0·22 | 0·33 | 0·21 | 0·48 |
| Free acid .. .... | 0·08 | 0·07 | 0·10 | 0·06 | 0·13 |
| Combined acid .. .. | 0·10 | 0·09 | 0·13 | 0·08 | 0·18 |
| Gums .. .. .... | 0·24 | 0·62 | 0·93 | 0·29 | 0·66 |
| Purity . .. .. .. | 89·07 | 82·53 | 82·53 | 81·38 | 81·38 |
| Glucose ratio.. .... | 5·92 | 5·32 | 5·32 | 6·03 | 6·03 |

The decrease in purity is caused by an increase of gums, albuminoids, acids, and ash in the second and third juices. In the experiment with steam-maceration, a jet of steam was introduced through the trash plates between the rollers of the second and third mills. The steam coagulated the albuminoids, so that less of these was removed than in the process of dry extraction; on the other hand, the percentage of gums in the juice was very much increased, so that the resulting juice was very difficult to clarify.

**Composition of Diffusion Juice.**

Diffusion juice does not show similar differences, firstly because only one juice is obtained, and secondly because but few cells are opened. The diffused juice, properly so called, is free from albumen and pectin, but as the slicing machine cuts through a certain number of cells, their contents mix with the juice, so that in practical working diffusion juice is sure to contain a small quantity of pectin, colouring matter, and albumen.

Keyer calculates that on slicing beetroots for diffusion, about 6·5 per cent. of the cells are opened and though such determinations have not yet been made with cane, the result will not be very different.

Further, the hot diffusion juice dissolves part of the gummy matters from the fibre especially when, as in Naudet's process, the bagasse is treated with hot limed juice. Neither enclosed air bubbles nor bagasse fibres are met with in the diffusion juice, while at the same time the amount of sand and clay carried along with the juice is also trifling. Another advantage of this juice is that it can be much more accurately measured, because the surface of the liquid is not covered with froth as mill juice usually is.

## 5.—Composition and Value of Bagasse as Fuel.

**Composition of the Bagasse.**

The bagasse (or megass as it is also called) coming from the mills contains an average of 46·5 per cent. fibre, 4·5 per cent. sucrose and 47·5 per cent. moisture, though, as observed in a previous chapter, higher or lower values may occur, depending on the quality of the crushing and the hardness of the cane fibre.

**Use of Bagasse for Fuel.**

Bagasse is generally used as fuel and in most cases it is sufficient to furnish all the steam required for driving the machinery and evaporating the juice in a modern factory turning out refining crystals. In the case of defective furnaces, or when making white sugar which requires more manipulation and consequently more steam, the bagasse is not sufficient and has to be supplemented by additional fuel, *e.g.*, cane trash, firewood or coal.

It is evident that the fibre-content of the cane and the density of the juice also come into account, as a high fibre content of the cane furnishes more bagasse than a low one, while juice having a high density will, as a rule, get more diluted by maceration than a poor one, which does not justify the use of much maceration water.

Besides the quantity of bagasse yielded by 100 parts of cane, its physical character also influences its calorific value.

**Calculated Calorific Value of the Bagasse.**

This latter can be calculated from the composition of any sample of bagasse and the calorific values of its constituents mentioned in Part I. after the following formula:—

$$C.V. = 8550\ F + 7119\ S + 6750\ G - 972\ W : 100$$

in which C.V. = calorific value in British Thermal Units for 1 lb. of bagasse, containing F per cent. fibre, S per cent. sucrose, G per cent. reducing sugar, and W per cent. water.

The calorific values of three different samples of bagasse were as follows (ash is put down as 3 per cent.):—

| | | | |
|---|---|---|---|
| F = 50 | S + G = 4·5 | W = 42·5 | C.V. = 4275 + 320·3 — 412·7 = 4182·6. |
| F = 40 | S + G = 6·0 | W = 51·0 | C.V. = 3420 + 427·1 — 495·7 = 3351·4. |
| F = 33·3 | S + G = 7·0 | W = 56·7 | C.V. = 2850 + 498·3 — 551·0 = 2797·3. |

An average Java bagasse, having 46·5 per cent. fibre, 4·50 per cent. sucrose, 0·5 per cent. glucose, and 47·5 per cent. moisture, possesses this calorific value:—

$$\frac{46{\cdot}5 \times 8550}{100} + \frac{4{\cdot}5 \times 7119}{100} + \frac{0{\cdot}5 \times 6750}{100} - \frac{47{\cdot}5 \times 972}{100} =$$

$$3975{\cdot}7 + 320{\cdot}3 + 33{\cdot}7 - 461{\cdot}7 = 3868 \text{ B.T.U.}$$

These figures show that the drier the bagasse is crushed the higher is its calorific value, although it contains less sucrose. Experts from other countries,

**Calorific Value of Bagasse is independent of its Sugar-content.**

where the bagasse is not so thoroughly exhausted as in Java, have frequently expressed surprise that so much steam can be produced by bagasse of so low a sucrose-content as these statistics show. The obvious reason is that, along with the sucrose, much water is simultaneously expressed. Residual juice contains sufficient organic substances (say 12 per cent.) to evaporate all the water present when burnt in a furnace.

$$\frac{12 \times 7119}{100} = 654{\cdot}3 \text{ and } \frac{(100-12) \times 972}{100} = 655{\cdot}4.$$

Therefore a juice containing 12 per cent. of sugar will be evaporated by the heat developed by the combustion of the contained sugar; and a bagasse containing such juice has a calorific value due only to the fibre contained in it, so that it makes no difference whether it be crushed dry or not. When the sucrose content of the juice is lower, the calorific value of the bagasse is increased by extracting more juice and, therefore, more sugar from it, and this explains why, with a high dilution by maceration and heavy pressure, the well exhausted Java bagasse can, in many instances, supply all the steam necessary in the factory without additional fuel being needed.

In these calculations it is, of course, assumed that during combustion of the organic matter in the bagasse only the highest products of oxidation are formed, but according to Bolk's* experiments, this requirement is not always fulfilled, so that the full calorific value is not really utilized. This is specially the case with bagasse, having a water content of over 50 per cent., because this does not burn properly but merely smoulders and produces a large quantity of products of destructive distillation, chiefly heavy hydrocarbons, which escape unburnt and are thus lost.

**Incomplete Combustion owing to excessive Moisture Content of Bagasse.**

Some canes with very soft fibre, and consequently yielding a very moist bagasse, were sent to the author for examination, because they would not burn but smouldered and eventually extinguished the fire. The engineers ascribed this to a high percentage of silica or of salt in the bagasse, but it soon became evident that the excessive amount of water accounted for the phenomenon. Sometimes the silica content was slightly higher than usual but the divergence never exceeded 0·5 per cent. and we cannot suppose that so trifling a rise in an infusible constituent like silica would render bagasse incombustible. The presence of soluble salts in the bagasse to an appreciable extent must also be rejected, for had they been present in the original canes in excessive quantities, they would have been removed with the sugar and the other soluble juice constituents by the different operations of mill-extraction and maceration, so that none would be found in the bagasse. In all those cases the author found a high water content; the bagasse did not actually burn but smouldered slowly and left

* *Archief voor de Java Suikerindustrie*, 1906, 319.

a black carbonaceous residue. The calorific value was greatly reduced and the engineers could not maintain the desired steam pressure in the boilers. At first sight one could only ascribe the deficiency in calorific value to the amount of unburnt carbon in the ash; but a simple calculation showed that this was immaterial and that the diminished heating power was due partly to the large amount of heat required to evaporate the surplus water and partly to the formation of products of destructive distillation which escaped unutilized through the flues. The blackest ash, containing the largest amount of carbon, was obtained from a bagasse with 52·50 per cent. of moisture; it contained 74 per cent. of inorganic matter, 21 per cent. of combustible matter, and 5 per cent. of moisture. The original bagasse, in its moist condition, had contained 46·38 per cent. of combustible matter, and 1·12 per cent. of ash. The original fuel therefore contained 1·12 parts of ash for every 46·38 of combustible matter, or 3091 of the latter for every 74 parts of ash. After combustion, we found only 21 parts of combustible matter to 74 parts of ash, which proves that this small amount of unburnt organic matter is of no real significance, when compared with the large amount which was burnt and disappeared in a gaseous form, and with the amount required to evaporate the water from the moist bagasse.

**Unburnt Organic Matter left in the Ash of Moist Bagasse.**

On some estates it was observed that the bagasse of some special cane variety yielded a more fusible ash than that of other varieties. Whereas the bagasse from certain varieties of cane gave no trouble in the furnaces and no clogged grate-bars, when other varieties were being crushed great care had to be taken to keep the grates free from clinkers, which hampered the draught and hindered the combustion. This, also, was often ascribed to the composition of the ash as containing more fusible constituents at one time than at another; but it soon appeared that the difference in the composition of the ash was imperceptible and that once again the physical condition of the fibre of the bagasse accounted for it. One kind of fibre can burn under conditions of draught and grate-area so different from those required by any other kind that in one case the ash fuses and in the other cannot reach such a high temperature. By adjusting the conditions of the furnace any bagasse can be burnt so as to yield a fused or an unfused ash. A good example of this is furnished by generator furnaces where such a high local temperature prevails that the bagasse-ash fuses into a glassy mass and clings to the walls of the furnaces in the forms of glassy lumps. The special construction of the furnaces keeps the grates free, so that no obstruction in the air supply is experienced. Now this same bagasse can be completely burnt in other furnaces without yielding a fused ash, which proves that it is not so much the character of the ash but that of the furnace which gives rise to the molten state. Indeed in the same furnace a flaming bagasse may yield a more fusible ash than a smouldering bagasse, and there is much more need to guard against extremes in either direction, by changing the slope of the grate or the space between its

bars, or similar expedients. If a bagasse leaves a fused ash, it is much better to see if some change cannot be made in the position of the grate than to continue scraping the grate bars at intervals.

**Diffusion Bagasse as Fuel.**

The bagasse resulting from the exhaustion of cane chips in the diffusion battery is pressed in mills and can theoretically be reduced to the same moisture content as milled bagasse. Experiments made by the author showed that bagasse which had soaked in hot water for hours had not absorbed more water than the cane fibre originally contained, so that it might be crushed as dry as ordinary megass by mill pressure. In practical working, however, the bagasse obtained on diffusion is so fine and the water content so high that the water cannot escape sufficiently freely from the mill, so is partly re-absorbed by the crushed bagasse. Only by passing the bagasse through two mills placed tandem (by which expedient the second mill is not obstructed by any large excess of water) can the moisture-content be reduced to 48 per cent.; but otherwise the water content is generally higher, especially if a large quantity of cane is worked up.

**Huillard's Bagasse Dryer.**

Instead of using a second mill, the surplus moisture may be removed from the fuel in a Huillard bagasse dryer.* This apparatus consists of a circular tower, the dimensions of which vary according to the quantity of material to be dried in a given time. On entering the top of the drier, the bagasse passes down a chute provided with valves to prevent the entrance of air, and thence falls upon the first horizontal partition near the circumference of same. It is here distributed by a revolving rake, which gradually draws it towards an outlet in the centre of the partition whence it falls on to a second partition, and so on, until it finally arrives at the ground level, whence it is removed by a carrier and conveyed to the furnaces.

The hot gases, leaving the boilers, are drawn upwards through the drier by a fan situated at the top of the tower, and as these gases pass from platform to platform they become more and more charged with aqueous vapour abstracted from the bagasse, and are finally discharged from the fan, either through a special chimney or through the chimney belonging to the boiler range.

The gases enter the drier at a temperature of about 250° C. and escape at the top in the form of a dense white cloud, having a temperature which varies between 50 and 60° C. The bagasse coming from the mill contains about 70 per cent. of water, and is dried in the Huillard apparatus to a water content of about 30 per cent.

According to calculations of Pellet,† even in the case of bagasse containing from 80 to 82 per cent. of water, the heat of the flue gases is sufficient to reduce the water content to between 30 and 35 per cent. when the whole of the bagasse

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* *International Sugar Journal*, 1907, 292. † *Internation l Sugar Journal*, 1907, 339.

produced is passed through the Huillard drier. It may therefore be said that under ordinary working conditions, the heat escaping up the chimney would suffice to dry the whole of the bagasse passing into the furnaces.

**Physical Condition of the Bagasse a Factor of its Fuel Value.**

Besides the chemical composition, the physical condition of the bagasse influences its fuel valve. It often happens that, for a given grate-area, sufficient steam is produced when hard canes are being crushed, but falls short as soon as soft canes pass through the mill. It soon appeared that this difference is due to variations in specific gravity of bagasse from different cane varieties as mentioned on page 49. Thus, in the case of hard canes, a greater weight of bagasse lies on the grate than in case of soft canes, and this alone is sufficient to affect the draught, the admission of air, and perhaps other circumstances also. Economic stoking may also be impossible in the case of some kinds of bagasse without making alterations in the furnace or in the grate-area.

**Molascuit.**

The use of bagasse for the manufacture of "molascuit" will be discussed at the end of this Section.

**Bagasse for Paper Making.**

Many attempts have been made to use bagasse as raw material for paper, and though the result was satisfactory in many cases, paper has not yet been produced from this material in large quantities. Owing to the comparatively small quantity of cellulose in cane fibre, the large percentage of lignin and the quantity of soda required to remove the latter, the cost price of such paper must be rather high. In countries such as Louisiana, where fuel and chemicals are cheap, and where there is an abundant supply of pure water for washing the pulp, the manufacture of paper from bagasse may have a good prospect before it, but for countries where chemicals and fuel are very expensive, or where good water is scarce, the prospects of this industry are doubtful. The manufacture of paper from bagasse was started some years ago on a sugar estate in Texas but had to be abandoned; but lately a successful enterprise has been reported from the Tacarigua Estate, Trinidad,* where very fine wrapping paper, that can also be printed on, is being manufactured from bagasse. This paper is, however, not made from bagasse alone, but from Para grass (15 per cent.), bamboo (20 per cent.), and bagasse (65 per cent.).

Dr. Viggo Drewsen succeeded at the Cumberland Mills, Maine, in making an excellent printing and letter paper solely from bagasse from Louisiana and Cuba. The average yield was 50 per cent., and the paper was prepared by heating the bagasse under pressure with a large amount of caustic soda.

But although the prospects are very promising, paper is not yet manufactured from bagasse on a commercial scale.

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* *International Sugar Journal*, 264.

CHAPTER II.

# CLARIFICATION.

## I.—Methods of Clarification.

The turbid, viscous juice from the mills is not fit to be worked up into sugar without clarification, and the first step is to submit the juice to such treatment as will remove the viscous and gummy constituents. As sucrose is very liable to decomposition by micro-organisms, which results in sourness, inversion and fermentation, and as cane juice is a very suitable medium for their development, it is necessary to boil the juice immediately, whereby the albuminoids are coagulated and the further development of the micro-organisms checked. It is also necessary to remove all impurities, both dissolved and suspended, so far as is practicable without spending too much on clarifying agents, which may cost more than the results are worth.

**Principle.**

The purpose of clarification is, therefore, to free the juice as quickly as possible from all constituents except sucrose, without alteration of the sucrose itself, and to perform the necessary alterations without delay since it is only in concentrated solution that sugar is unaffected by ferments and micro-organisms.

Besides heat, which coagulates the albumen and kills the germs, the agent which has been used for clarification from the earliest times is *lime*. This precipitates gummy and albuminoid matters which form a muddy sediment, and a top layer of scum, between which the bulk of the juice is clear and limpid. (*See below.*)

**Agents Employed in Clarification.**

The methods of clarification may be divided into two classes, viz., defecation and carbonatation, either of which may be applied in various ways. In defecation only a small quantity of lime is used, so that very little or no excess of lime remains after clarification, while in the carbonatation process the juice is heavily limed and the excess afterwards removed by carbonic acid.

### DEFECATION.

#### A.—Treatment of the Raw Juice.

The quantity of lime used in the defecation process is just sufficient to neutralize the free acids of the juice, preventing them from afterwards causing inversion; further, the lime combines with the albumen, which had previously been suspended in the juice in a gelatinous form, but is now

**Principle.**

coagulated both by the action of lime and of heat. The acid calcium phosphates are precipitated as flocculent tribasic phosphate, as well as a part of the pectin. All these flocculent precipitates envelop the suspended particles of bagasse, the wax, clay, &c., and rise to the surface as a thick scum as soon as the juice begins to boil.

**Tempering.**

The estimation of the quantity of lime necessary for clarification is the most important point in the defecation process. When too small a quantity has been added, the phosphoric acid, the gummy matters, and the albumen are imperfectly precipitated, and the sediment settles slowly. When too much lime is used, the impurities are easily precipitated, and settle quickly. But another difficulty then arises. Lime, like other alkaline agents, acts on the reducing sugar, at the high temperature at which the defecation is conducted, forming lime salts of organic acids, which are dark-coloured and easily decompose to form acid substances which cause inversion of the sugar. Moreover, these lime salts retard evaporation and crystallization, and their viscosity causes considerable loss on curing. The greatest care is therefore advisable, and if there remains a doubt as to whether the right quantity of lime is being used, it is better to add a little too much rather than too little, for in an insufficiently tempered juice the impurities still remaining cause much trouble during the whole course of manufacture, while an over-tempered one may always be neutralized by phosphoric or sulphurous acid.

**Determination of the proper Amount of Lime for Tempering.**

The amount of lime necessary for tempering is ascertained in different ways. Some use as much lime as is necessary for the juice to give a faint alkaline reaction on litmus or turmeric paper; others ascertain in the laboratory how many c.c. of lime-milk are sufficient to clarify one litre of juice at its boiling point, and use that proportion in the factory. The best method is as follows: First, as much lime is added to the juice as seems appropriate, say two gallons of milk of lime of 15° B. to 500 gallons of juice, and clarification is effected as usual. Then a little juice is taken from the clarifiers, filtered through paper into a test tube, and a few drops of sucrate of lime solution added.* If a precipitate is formed, it is evident that the juice still contains impurities, and that therefore too little lime has been used. In the next clarifier a somewhat larger quantity of milk of lime is used in tempering, the juice is once more tested, and if it becomes turbid with the sucrate of lime solution, the quantity of lime is again increased until the filtered juice remains clear after the addition of the test solution. If, on the contrary, the sucrate of lime leaves the juice clear at the first test, either just sufficient or too much lime has been added to the juice. In order to settle this point we gradually diminish the quantity of lime in tempering until the juice becomes under-tempered, when the amount of lime is slightly increased, and this quantity adopted as correct.

* This solution is made by shaking up lime with a 20 per cent. sucrose solution, and filtering. It is much to be preferred to lime water, because a slight precipitation can be better observed with this test solution than with lime water.

This method seems rather complicated, but after some experience the proper quantity is soon found with a few tests. As the chief aim of clarification is to get a juice that clarifies easily, this method of determining the necessary quantity of lime is the most rational one, and if so much lime is required as to make the juice alkaline after clarification, it may afterwards be neutralized by phosphoric or sulphurous acid. When making high-class sugar a slightly acid reaction of the juice is even an advantage, but in making the so-called "non-chemical" sugar, the juice should be neutral or even a little alkaline, for in these sugars a rather considerable amount of molasses remains between the sugar crystals, which, in the case of acidity of the juice, gives rise to inversion and deterioration of the sugar.

**Difficulties in Tempering occasioned by varying Composition of the Juice.**

A great difficulty in the proper control of the clarification is the varying composition of the mill juice when canes from different fields are being crushed at the same time. The juice of the canes of each field requires its own special quantity of lime, and if canes of different fields are crushed together, the composition of the mill juice varies every moment, and hence also the quantity of lime necessary for clarification. Of course it is impossible to determine it for each clarifier, and therefore defecation is a difficult and somewhat uncertain operation.

The mill juice is filtered through copper strainers, and measured in measuring tanks where it is limed; it next passes through a juice-heater, and then enters the clarifiers.

**Dextran Fermentation.**

Should the juice be infected with *Leuconostoc mesenterioides*, it is advisable to heat the juice before liming it. The *Leuconostoc* grows very rapidly in alkaline sugar solutions and only very slowly in neutral or acid ones. When, therefore, the cold juice is limed in the measuring tanks and flows through pipes, cocks, or valves, even a slight infection with *Leuconostoc* may cause these to become choked. A small deposit of dextran in such places is apt to grow very rapidly in the current of alkaline juice. On the other hand, the growth of *Leuconostoc* is soon checked in a small tank because this organism produces acid substances which are detrimental to itself. But, when growing in a pipe or other locality through which alkaline juice is constantly passing, the liquid never becomes neutral or acid, and the development of the organism is incredibly rapid. Instances where large pipes have been speedily choked would surprise those who witness it for the first time. It is evident that in unlimed (acid) juice the formation of dextran cannot proceed, and there is consequently no fear of clogging the bores of cocks or valves. In cases of infection with *Leuconostoc* it is therefore advisable to pump the unlimed juice through juice-heaters and thus kill the germs of *Leuconostoc*, and to lime the juice afterwards. Experiments on this point proved that cane juice may safely be heated for a few minutes to 100° C. without appreciable inversion.

**Sulphitation of Limed Cane Juice.**

Some juices require so much lime for the complete precipitation of the gummy and pectin matters that it becomes strongly alkaline after tempering and requires neutralizing. Besides, when making high grade sugar, a more thorough clarification is desirable, yet without showing an alkaline reaction. In order to obtain this we can combine sulphitation with the usual defecation process. In order to rapidly precipitate the gummy constituents, the cold juice is heavily limed in the measuring tanks, as much as 1 to 1½ gallons of lime cream of 15° Beaumé being added per 500 gallons of juice in excess of the quantity found necessary by the test mentioned on page 130. The cold juice is then pumped to the sulphur box, which is a vertical wooden chamber provided with perforated plates. The juice enters at the top and flows through the apertures in the plates in a continual stream, while the sulphur fumes enter the box at the bottom, impregnate the falling juice, and escape at the top. A better plan is as follows: Large iron tanks are filled with the cold limed juice, and sulphurous acid is blown into it through leaden pipes provided with rubber check valves. The supply of the acid is continued until a drop of the juice placed on phenolphthalein paper causes only a faint pink coloration instead of a deep crimson. The supply of sulphurous acid is then stopped, and the neutral juice pumped to the juice heaters without subsiding or skimming. This heavy liming and neutralization effects a more perfect clarification, with diminished viscosity, so that the subsequent separation of the molasses from the crystals is easier than when the juice has been tempered with just the required amount of lime. Caution must be used when neutralizing, and on no account should limed cane juice be heated before it is so far neutralized as only to show a slight alkalinity, otherwise it will become dark-coloured and cannot yield white sugar.

**Scaling of the Juice Heaters.**

The juice heaters rapidly become incrustated by the sulphured juice, but this causes little delay when duplicate heaters are available, one of which may be scraped clean while the other is in use. Such scale has the following composition:—

| | |
|---|---|
| Silica | 21·12 |
| Sulphuric acid | 7·66 |
| Sulphurous acid | 5·05 |
| Phosphoric acid | 3·78 |
| Iron oxide | 1·33 |
| Lime | 16·33 |
| Organic matter | 34·75 |
| Water | 8·25 |
| Undetermined | 1·73 |
| | 100· |

**Choking of Juice Heaters.** When sand or clay adheres to the rind of the cane, or when the cane is covered with volcanic ash or dust (as is sometimes the case), the juice heaters may become choked, and a settling tank should therefore be interposed between the mills or measuring tanks and the juice heater; but this is usually unnecessary.

**Clarification by the Old Method.** The defecation may be conducted in two ways, depending on the manner in which the precipitated impurities separate from the juice. In the first method the juice is allowed to subside in the same vessel in which it is heated, while in the other the limed and heated juice is run into settling tanks.

When adopting the first method the juice is strained through copper gauze screens, measured and limed in the measuring tanks, passed through juice heaters, which raise its temperature to about 80° C., finally entering the defecators, where it is further heated to the boiling point.

**Defecators.** The defecators are hemispherical copper pans with steam-jackets into which steam is admitted during the time that the juice fills the pans. The precipitated impurities coagulate, envelop the suspended particles, and air bubbles escaping from the hot juice, forming a frothy, dark-coloured layer of scums, underneath which is the clear juice. Only the heavy impurities, such as sand, clay, &c., sink to the bottom and form another layer, so that the juice is separated into three layers, of which the middle one, consisting of clarified juice, is the greatest. The juice is heated until the layer of scum begins to burst (cracking point); the supply of steam is then shut off, for if not, the juice would boil over, and be partly lost. The juice is now allowed to settle, and afterwards let off through a hole at the bottom of the pan, which is fitted with a pipe closed by a three-way cock. By turning the plug one is able to draw off successively the heavy impurities, the clean juice, and finally the scum, each of these being conducted into its own gutter. The first and third portions, *i.e.*, the heavy impurities and the scums, are run into the scum tank, while the clean juice is conducted to the evaporators or to the eliminating pans. The three-way cock and the gutters should be placed in an easily accessible and well-lighted position, so as to admit of complete control. In order to facilitate the decanting and the separation of the clear liquid from the mud and scums, some defecators have two outlets at the bottom, one for the sediment and the other for the clear juice: the latter carrying a pipe which extends above the surface level of the sediment within the tank. When the defecation and subsidation are completed, the latter cock is opened and the clarified juice discharged, without risk of contaminating same with the subsided mud, which remains below the outlet level of this pipe. The clarified juice is allowed to flow out until the scums approach the open end of the pipe, whereupon the cock is closed and the other discharge pipe opened, when the subsided mud, scum, and

**Settling and Decanting.**

thin layer of clarified juice between these two, passes into the scum tank for further treatment.

In the other method, the limed juice, after having passed through a juice-heater, is boiled in iron pans, through which steam is led by means of coils. As soon as the juice boils a cock is opened at the bottom of the pan, and the hot juice flows into the settling tanks, where the separation of the clear juice from the scum takes place. As in this case the air has been completely driven out by the ebullition, the scum does not remain as a froth on the surface, but settles gradually, leaving the juice quite clear.

**Clarification by the New Method.**

Most factories are provided with special cylindrical iron pans having broad gutters. In these pans a steam chamber is suspended, reaching nearly to the bottom. This is perforated by a number of tubes in which the juice circulates, and is thus caused to boil rapidly.

**Pans.**

As the frothy scum rises to the surface, it is driven into the gutters by long skimmers, whence it flows into the scum tank. When no dark coloured particles are visible at the surface, but only a white mass of steam bubbles, the steam supply is stopped and the juice with the heavy impurities is let off into the settling tanks.

In many places the juice is heated to about 90° in the juice-heater, and then allowed to flow through one or more pans, in which it boils rapidly, and thence passes into the settling tanks in a continuous stream. In this case no skimming can take place, but as the boiling breaks up the layer of scums, this mixes with the heavier deposits and does not interfere with the subsiding in the settling tanks. The latter are square iron tanks, covered externally with wood to retain the heat, and provided with cocks at varying distances from the bottom. The juice settles, the uppermost cock is opened and afterwards the others, so that only the clear juice flows off and goes to the clarified juice tank. Finally a cock at the bottom is opened, and the scum flows out and is run into the scum tank.

**Continuous Defecation.**

**Settling Tanks.**

Instead of a row of cocks, we often find a single cock connected with a hinged copper pipe, the free end of which is fixed to a hollow copper ball which floats on the juice, so that only the uppermost and clearest layer of the juice passes through the pipe.

When the juice has a high density, and the quantity of impurities is small, it often happens that the small amount of scum remains suspended; settling may then be promoted in two ways. Some pour cold water from an ordinary watering can upon the hot juice, which dilutes the juice at the surface and causes it to throw down the impurities; and further a downward stream is produced by the cold water, which also imparts a downward direction to the scum.

**Difficulties in Subsiding and their Remedies.**

If subsidation cannot be brought about by a longer stay in the clarifiers, the scum may be rendered heavier and more flocculent by using a little more lime in tempering and adding the quantity of phosphoric acid necessary to neutralize this excess. The amount of dissolved lime remains the same, but a heavy, flocculent precipitate of tribasic phosphate of lime is produced, which envelopes the fine particles of scum, and carries them to the bottom.

It is not advisable to pump the juice through the juice heaters too quickly, as it frequently happens that the time during which the juice is heated is not sufficient to allow of complete defecation. Even when using very powerful juice-heaters one should not omit to boil the juice in a defecator before it is discharged into the settling tanks. To promote subsidation it has been proposed to add an emulsion of clay and water, but this has not found general application.

Barium aluminate has been recommended during the last few years for promoting subsidation. The barium combines with the sulphuric acid of the sulphates to form barium sulphate while the flocculent alumina carries down the floating impurities, leaving the juice clear. Up to the present the author has not heard of this chemical agent being used anywhere.

**Deming's System of Clarification.**

A third method of defecation, devised by Deming, has recently come to the front, in which the limed juice is superheated and subsequently passed through continuous subsiders. The limed juice is pumped through three vessels, in which it is heated under pressure to over 110° C., and afterwards cooled to about 100° C., so that the juice entering the apparatus at the ordinary temperature is heated for about 45 seconds at a temperature of 110-115° C., and leaves it at a temperature of 95-100° C. The three vessels are horizontal cylinders, in which tubes of 2in. diameter are fixed and connected with each other by chambers at either end of the cylinders in such a way that the juice flows backwards and forwards through the tubes with great velocity. Two of the vessels are called "absorbers"; the third (having the largest dimensions) bearing the name of "digester." In the first absorber the cold juice is introduced outside the tubes, but passes through the tubes of the second absorber, around which exhaust steam is circulated. Afterwards the juice flows through the tubes of the digester, is heated by live steam to the required temperature of 110-115° C., and finally passes through the tubes of the first absorber, where it imparts its superheat to the fresh juice surrounding the tubes, heating it to 40-50° C.

**Scale on the Outside of the Tubes of a Deming Apparatus.**

Owing to the high velocity with which the juice is forced through the tubes there is no fear of scaling; in fact they remain quite clean after some months' use. The apparatus only now and then requires a thorough cleaning with hot dilute hydrochloric acid, more especially the outside of the tubes in the first absorber, which become coated with mud from the cane juice.

An analysis of the air-dried scale from the outside of the tubes gave these figures:—

| | | |
|---|---|---|
| | Moisture | 4·66 |
| | Organic matter | 52·56 |
| In the Ash. | Insoluble in hydrochloric acid | 9·12 |
| | Lime | 14·22 |
| | Magnesia | 4·95 |
| | Iron oxide | 2·18 |
| | Alumina | 0·65 |
| | Phosphoric acid | 1·92 |
| | Sulphuric ,, | 0·27 |
| | Carbonic ,, | 8·20 |
| | Undetermined | 1·27 |
| | | 100·— |

The scale, therefore, consisted of calcium phosphate, other lime combinations with organic matter (acids, albumen, pectin), which changed into calcium carbonate during combustion, sand, clay, and partially caramelized constituents of the cane juice, which had become overheated by contact with the tubes.

**Settling.**

The hot juice is conveyed into settling tanks whence it is continuously discharged. These tanks are cylindrical with conical bottoms, carrying wide discharge tubes provided with a cock. A large cone of sheet iron (open at top and bottom) is suspended in the cylinder, the height of the cone being equal to that of the cylinder, so that their upper edges are at the same level and the bottom of the cone level with the conical part of the settling tank; the distance between the cone and the tank at that place being slightly over one inch.

The heated juice enters the tank outside the cone, fills it and overflows through a wide tube leading out of the upper part of the cone. This arrangement causes a downward current of juice outside, and an upward current inside the cone. The scum has therefore a tendency to sink to the bottom of the tank, and if it is induced to rise by the upward current inside the cone it is caught by the inclined side of the cone and again sinks. Care should be taken frequently to discharge subsided scum, so that no layer of dirt forms between the edge of the cone and the cylinder, as in that case lumps of scum may be carried to the surface and spoil the clarified juice.

In the case of slowly settling juice, the same expedient may be used as in the case of the ordinary clarification, *i.e.*, rendering the precipitate heavier by adding more lime and a corresponding amount of phosphoric acid to the mill juice.

Ordinarily, two settling tanks are required, the juice flows first through the smaller, afterwards through the larger one.

This system of clarification divides the juice continuously into clarified juice, ready for evaporation, and scum, which is conveyed to the scum tank.

**Advantages and Disadvantages of the different Methods of Clarification.**

Each method of defecation has its advantages and its defects. The advantage of the first method is that the juice is not heated to its boiling point, hence steam is saved, and, further, no danger of inversion or decomposition is to be feared. A drawback, however, is the great number of expensive defecators required if a properly clarified juice is to be obtained.

The common defecation pans with coils are easy to clean, which is not the case with the pans in which the juice is boiled in tubes of small diameter, unless a sufficient number of these are available, enabling us to spare one for cleaning, and go on working with the others. The skimming off of the light floating impurities has the disadvantage of keeping the juice for a long time at a high temperature, involving extra expenditure of steam, and possible decomposition of a portion of the sugar; though, as will be shown later, this danger is not a serious one.

Against these disadvantages must be set the prompt removal of the lighter portions of the scums in the skimmings, leaving only the heavier and more quickly subsiding portion to settle in the tanks, which saves much time, and increases the working capacity at this stage.

**Comparison of the Juices obtained by the ordinary Defecation and by Deming's Superheat Defecation.**

The advantages of superheat clarification are economy of steam, labour, and space, and that superheated juice subsides better than boiled juice. No chemical difference between juice clarified by the two ways can be detected, as is shown by the following analyses of juice from cane of the same field, clarified by the usual clarification, and by Deming's method on alternate days.

First Day.

Variety of cane .. .. .. .. .. .. .. .. Cheribon.
Defecation process .. .. .. .. .. .. Old defecation.
Liming .. 4 to 4½ litres lime 15° Beaumé to 1000 litres juice.

| | Brix. | Polarization. | Quotient. | Glucose. | Glucose ratio. |
|---|---|---|---|---|---|
| First mill juice .. .. .. .. .. | 16·93 | 14·97 | 88·40 | 1·15 | 7·7 |
| Last mill juice.. .. .. .. .. | 10·58 | 8·86 | 83·70 | 0·70 | 7·9 |
| Mixed juice .. .. .. .. .. | 14·47 | 12·69 | 87·70 | 0·98 | 7·7 |
| Clarified juice .. .. .. .. .. | 14·87 | 13·21 | 88·90 | 0·90 | 7·4 |
| Syrup .. .. .. .. .. .. .. | 50·59 | 44·90 | 88·70 | 2·90 | 6·5 |
| Masse cuite.. .. .. .. .. .. | 91·72 | 81·22 | 88·50 | 5·89 | 7·2 |
| First molasses .. .. .. .. .. | 79·52 | 49·20 | 61·86 | 14·24 | — |

SECOND DAY.

Variety of cane .. .. .. .. .. .. .. .. Cheribon.
Defecation process .. .... Deming's superheat clarification.
Liming .. .. .. .. .. .. $4\frac{1}{4}$ litres to 1000 litres juice.

| | Brix. | Polarization. | Quotient. | Glucose. | Glucose ratio. |
|---|---|---|---|---|---|
| First mill juice .. .. .. .. .. | 17·35 | 15·27 | 88·00 | 1·24 | 8·0 |
| Last mill juice.. .. .. .. .. | 9·02 | 7·70 | 85·30 | 0·60 | 7·8 |
| Mixed juice .. .. .. .. .. .. | 14·55 | 12·55 | 86·20 | 1·00 | 8·0 |
| Clarified juice .. .. .. .. .. | 14·72 | 12·67 | 86·10 | 0·94 | 7·4 |
| Syrup .. .. .. .. .. .. .. | 37·98 | 33·40 | 87·90 | 2·58 | 7·7 |
| Masse cuite.. .. .. .. .. | 90·30 | 80·12 | 88·70 | 6·05 | 7·5 |
| First molasses .. .. .. .. .. | 80·92 | 50·60 | 62·54 | 16·25 | — |

THIRD DAY.

Variety of cane .. .. .. .. .. .. .. .. Muntok.
Defecation process .. .. .. .. .. .. Old defecation.
Liming .. .. .. .. .. 4 to $4\frac{1}{2}$ litres to 1000 litres juice.

| | Brix. | Polarization. | Quotient. | Glucose. | Glucose ratio. |
|---|---|---|---|---|---|
| First mill juice .. .. .. .. .. | 18·70 | 16·88 | 90·3 | 1·24 | 7·4 |
| Last mill juice.. .. .. .. .. | 9·30 | 7·84 | 84·3 | 0·57 | 7·3 |
| Mixed juice .. .. .. .. .. .. | 16·10 | 14·23 | 88·4 | 1·03 | 7·2 |
| Clarified juice .. .. .. .. .. | 16·53 | 15·00 | 90·8 | 0·99 | 6·6 |
| Syrup .. .. .. .. .. .. .. | 45·72 | 41·2 | 90·1 | 2·56 | 6·2 |
| Masse cuite.. .. .. .. .. .. | 92·00 | 82·94 | 90·2 | 5·42 | 6·5 |
| First molasses .. .. .. .. .. | 79·6 | 50·8 | 63·8 | 14·80 | — |

FOURTH DAY.

Variety of cane .. .. .. .. .. .. .. .. Muntok.
Defecation process .. .... Deming's superheat clarification.
Liming .. .. .. .. .. .. 4·2 litres to 1000 litres juice.

| | Brix. | Polarization. | Quotient. | Glucose. | Glucose ratio. |
|---|---|---|---|---|---|
| First mill juice .. .. .. .. .. | 18·71 | 16·72 | 89·40 | 1·53 | 9·1 |
| Last mill juice.. .. .. .. .. | 10·70 | 9·02 | 84·30 | 0·84 | 9·3 |
| Mixed juice .. .. .. .. .. .. | 16·92 | 14·71 | 87·00 | 1·37 | 9·3 |
| Clarified juice .. .. .. .. .. | 16·67 | 14·69 | 88·10 | 1·21 | 8·2 |
| Syrup .. .. .. .. .. .. .. | 40·7 | 36·40 | 89·40 | 3·02 | 8·3 |
| Masse cuite.. .. .. .. .. .. | 91·4 | 80·51 | 88·10 | 6·63 | 8·2 |
| First molasses .. .. .. .. .. | 79·8 | 48·80 | 61·03 | 16·00 | — |

### B.—Treatment of the Clarified Juice.

In many factories the juice, after settling, is pumped without any further treatment into the suction tank of the evaporators, in others it passes through one or other form of filter, in others it is once more boiled in eliminating pans of the shape previously described. This treatment is especially necessary for juice clarified by the first-named method, as a great part of the scums often accompanies the clarified juice owing to want of attention. The juice, after settling, is boiled, skimmed, rendered neutral by phosphoric or sulphurous acid if the juice is too alkaline, or with soda or lime if it is too acid, because, as already mentioned, a neutral or very slightly acid or alkaline juice is the most advantageous.

Juices which have been allowed to settle in special clarifiers do not need elimination, for in this case the juice can be neutralized directly in the defecators.

If the juice is too acid, a solution of caustic soda or carbonate of soda of 20° Beaumé is added in the case of "non-chemical sugar" until the juice shows a slightly alkaline reaction to litmus paper. If, on the other hand, the juice is too alkaline or *neutral* and it is intended to make high-class sugar, the juice is rendered faintly acid with a solution of phosphoric acid of 20° Beaumé or a stream of sulphurous acid gas is conducted into the juice until the proper acid reaction is obtained.

### C.—Treatment of the Scums.

The scums from the clarifiers, or from the settling tanks, are run into scum tanks as already mentioned. Sometimes the scums are allowed to settle once more, and the clear juice is again drawn off, but generally the scums are mixed with lime and steam to promote filtration. Many factories only possess a single scum tank, into which the scum continually flows from the clarifiers; it is heated and limed from time to time, and pumped off to the filters, but in this way it is impossible to know whether the scum is sufficiently limed or not. It is much better to have two tanks, and use them alternately, so that the first can be properly limed, steamed and emptied, while the second is being filled. Instead of this, a mixer can be put in front of the steaming tank in which the scums are limed.

If the juice has been properly tempered and at a proper temperature, the scums can be filtered without further addition of lime; but this helps if clarification has been defective.

### D.—Influence of Defecation on the Constituents of the Juice.

The quantity of impurities separated from the juice by defecation is not considerable, since the weight of wet mud (chiefly consisting of juice) does not amount to more than one per cent. of the weight of the cane.

**Composition of Defecation Mud.** The composition of the mud is as follows :—

| Constituents. | Taylor Filter. | Taylor Filter. | Filter Press. | Filter Press. |
|---|---|---|---|---|
| Moisture | 72·14 | 67·73 | 69·26 | 69·72 |
| Sucrose | 13·20 | 10·50 | 9·96 | 10·20 |
| Reducing Sugar | 0·60 | — | — | 0·71 |
| Ash | — | — | 7·59 | 8·10 |
| Albuminoids | 1·93 | 0·87 | 1·94 | 1·80 |
| Wax | — | 3·04 | 5·13 | 4·12 |
| Fibre | — | 11·00 | — | 5·35 |
| Lime | 0·66 | 0·67 | — | 1·53 |
| Magnesia | — | 0·08 | — | 0·22 |
| Iron Oxide and Aluminia | — | 3·06 | — | 1·40 |
| Phosphoric Acid | 0·57 | 1·49 | 1·42 | 1·39 |
| Silica | — | 0·21 | — | 0·37 |
| Sand and Clay | 1·38 | 1·70 | — | 2·82 |

We may assume the amount of dry substance in the mud to be 30 per cent., of which 10 per cent. are sugars and 11 per cent. are total dissolved matters. In the ash we find about 1·50 per cent. of lime, so that about 17·50 per cent. of the weight of the moist filter press mud consists of soluble and suspended impurities removed from the juice. On an average we obtain, say, 1·10 per cent. of the weight of cane in the form of filter mud, thus defecation decreases the percentage of non-sugars in the juice by ± 0·2 on 100 parts of cane. The specific gravity of the fibre being about 1·0, that of sand and clay 2·5, and that of calcium phosphate 2·8, the total specific gravity of these impurities will not differ much from that of sucrose, and we are therefore justified in assuming that for every 100 parts of cane (or diluted juice) 0·2 degrees Brix of solid matters, more or less, pass into the filter press mud.

**Amount of Dry Substance Removed from Cane Juice during Defecation.**

At a Brix of 16 and purity of 85, these 0·2° Brix cause a rise in the quotient of purity of about one point; at a higher Brix and a lower purity this figure is less, but the difference is so insignificant, that we may accept 1 point as the increase in purity, due to the removal of non-sugars from the juice by the defecation process.

**Calculated Increase of Purity by Defecation**

The chemical control in Java sugar houses during 1905 showed an average increase in purity of 1·7, in 1906 1·6, and in 1907 1·9, or more than the calculated figure, while the individual differences apart from extreme cases are not considerable. From these figures it may be concluded that defecation does more than merely precipitate impurities, but we must bear in mind that this increase in purity may be more apparent than real since

**Apparent Increase of Purity by Defecation.**

the degrees Brix do not strictly represent the dry substance, nor the polarization the true sucrose content. Other bodies present in the juice also possess rotatory power, which may vary in acid and alkaline solutions, thereby affecting the purity, as such changes in rotatory power of one constituent might easily do.

**Abnormally High Purity of some Cane Juices.**

In some cases an abnormally high purity of the raw juice falls to the normal figure after defecation. Winter* describes a number of such cases observed at Kaliwoengoe estate in Java, while no constituent could be detected which accounted for this phenomenon.

In most cases the purity of the mixed juice was higher than that of the first mill juice, contrary to general experience. We know of amids and amido acids, having high rotatory powers when treated with basic lead acetate, but such were not found in sufficient proportions to explain so considerable an increase in the rotatory power; moreover, when the juice was clarified with reagents other than lead compounds, it showed a similar abnormal purity. But after defecation and evaporation, the purity fell and the syrup showed quite a normal composition.

**An abnormal Purity becomes Normal after Clarification and Evaporation.**

A series of analyses of the same juice at different periods of manufacture may illustrate this point.

| | Brix. | Polarization in the 200 mm. tube. | Calculated Sucrose. | Purity. |
|---|---|---|---|---|
| First mill juice .. .. .. .. | 20·3 | 73·2 | 19·30 | 95·07 |
| Second mill juice .. .. .... | 19·66 | 69·3 | 18·35 | 93·15 |
| Third mill juice .. .. .. .. | 7·7 | 25·1 | 6·97 | 90·0 |
| Mixed juice.. .. .. .. .... | 17·4 | 62·2 | 16·58 | 95·40 |
| Clarified juice.. .. .. .. .. | 17·0 | 60·2 | 16·1 | 94·59 |
| Syrup .. .. .. .. .. .... | 63·8 | 58·8 | 58·8 | 92·0 |

The purity diminished by 1 point on defecation, and by 2·5 additional points during evaporation, notwithstanding the fact that non-sugars were removed during defecation. The author repeatedly sought to explain this phenomenon by the presence of dextro-rotatory impurities until it occurred to him that the degrees Brix might be an unreliable measure of the solids in solution and thus account for the abnormal purity. If the cane juice contains a volatile substance of less specific gravity than water, the specific gravity and therefore the degrees Brix of the juice would be less than that due to the solids in solution and the purity accordingly too high. When such juice is evaporated, the volatile constituent would disappear, leaving the syrup with a normal specific gravity and quotient of purity. The author's hypothesis† was that juices from damaged or

* *Uit het chemische Laboratorium*, 87. † *International Sugar Journal*, 1907.

diseased cane might contain alcohol, which by reason of its lesser specific gravity and volatility could produce the observed phenomenon, but H. Pellet pointed out that in that case the juice must have shown other irregularities. He agrees that some light and volatile body is the cause of the anomaly, but thinks that this may be imprisoned air. Juices containing much albumen or pectine are very liable to become emulsified, the air being so firmly held that it does not escape even after standing a couple of hours. The specific gravity of this emulsion being of course less than that of the juice freed from air, the calculated purity is too high when based on the degrees Brix. Heat and evaporation expel the air, and thus restore the normal specific gravity. Pellet's theory also explains why the purity of the mixed juice is higher than that of any one mill alone, namely, the agitation of the combined juices in the mixing tank which tends to increase the state of emulsion. This fact could not be explained according to the author's theory.

In other cases the purity of the last mill juice was higher than that of the other mills, and sometimes the wash-waters from the filter press showed an unusually high quotient, so that the possibility is not excluded that the fibrous part of the cane occasionally contains some dextro-rotating substance, this being removed by defecation but partially redissolved on steaming the scums.

Unless the quotient of purity of the mixed mill juice be based on the actual dissolved solids (instead of on degrees Brix), we should not attach much value to the decrease or increase of purity during defecation. An increase of less than one point is not necessarily an indication of inversion or decomposition, while on the other hand a greater increase is not a sign of more perfect clarification of the juice.

**Effects of Defecation on the separate Juices obtained with Triple-Crushing.**

The effects of the defecation on the constituents of each of the three mill juices is represented in the following table:—

| Constituents. | First mill. | | Second mill. | | Third mill. | | |
|---|---|---|---|---|---|---|---|
| | Before. | After. | Before. | After. | Before. | After. | |
| | | | | | | Little lime. | Much lime. |
| Brix .. .. .... | 19·2 | 19·2 | 19·3 | 19·7 | 19·0 | 19·4 | 19·2 |
| Sucrose .. .. .. | 16·49 | 17·06 | 16·33 | 17·06 | 15·95 | 16·40 | 16·50 |
| Reducing Sugar .. | 1·98 | 2 | 1·57 | 1·58 | 1·52 | 1·52 | 1·41 |
| Gums and Pectin .. | 0·125 | 0·071 | 0·376 | 0·120 | 1·250 | 0·840 | 0·38 |
| Albumen .. .... | 0·025 | — | 0·092 | — | 0·054 | — | — |
| Quotient of Purity. | 85·9 | 87·5 | 84·4 | 86·6 | 84· | 84·5 | 86·0 |
| Glucose Ratio .... | 12·1 | 11·7 | 9·6 | 9·2 | 9·5 | 9·3 | 8·6 |

**Water.** The loss of water during defecation is insignificant when hemispherical pans or the closed Deming's apparatus are used, but with continuous defecation without skimming the loss may be put down as 1·5 per cent., and with skimming, as 3 per cent.

Exposure to high temperatures always inverts some sucrose, and the minimum loss may be calculated from Herzfeld's tables mentioned in Part I., Chapter I.

**Sucrose.**

When the juice is sour, or partially inverted, the losses are of course larger, but so long as the work is conducted regularly without stoppages losses of sucrose due to inversion or sourness need not be feared.

| | Time during which the juice stands. Minutes. | Average Temperature. | Loss of Sucrose per hour. | Total loss on 100 Sucrose. |
|---|---|---|---|---|
| Defecators .. .. .. .. .. .. .. | 15 | 100° | 0·0961 | 0·0240 |
| Settling tanks.. .. .. .. .. .... | 100 | 80° | 0·0373 | 0·0622 |
| Receiving tank for the clarified juice. | 30 | 80° | 0·0373 | 0·0182 |
| | 145 | | | 0·1044 |

We have already mentioned in a previous chapter that at high temperatures lime acts upon the reducing sugars with the formation of lime salts of organic

**Reducing Sugar.**

acids, the remainder of the reducing sugars being a mixture of glucose, fructose, and mannose, and optically inactive. Consequently, an excess of lime (sufficient to cause an alkaline reaction of the clarified juice) will first destroy a small part of the total reducing sugars (thereby diminishing the glucose-ratio) and, further, will destroy the levo-rotation of the balance of the reducing sugars—thereby increasing the dextro-rotation attributed to sucrose. Both of these influences, therefore, tend to increase the apparent purity of the juice.

The proportion of lime salts in the juice will become greater than the sum of the lime pre-existing in the juice and of that which has neutralized the free

**Action of the Lime.**

acid in the juice. In cases where unnecessarily large amounts of lime-cream are used in tempering, all these phenomena certainly occur, namely, a fall in the glucose-ratio, a rise in the purity, and an unusually high lime content in the syrups and molasses. A second consequence of over-tempering is spontaneous decomposition of the organic lime salts, causing the final masse cuites to foam with the escaping gaseous products of decomposition. These are, however, exceptions, and generally the juices are tempered with sufficient care.

The following table shows that the rotatory power of the reducing sugar is not appreciably affected during defecation. As has been observed on page 84

**Change in Rotatory Power of the Reducing Sugar by Defecation.**

trifling errors in analysis are sufficient to cause considerable deviation in the calculated rotatory powers, so that even a difference of five points is well within the limits of error. Taking this into consideration, we see that the rotations of the reducing sugars are not much influenced by defecation.

In this table the analyses of juice and first molasses are bracketed together as referring to the same factory.

| | Brix. | Polarization before Inversion. | Polarization after Inversion. | Calculated Rotation of the Sucrose. | Difference. | Reducing Sugar. | Rotatory Power of the Reducing Sugar. | Sucrose. | Glucose. | Fructose. |
|---|---|---|---|---|---|---|---|---|---|---|
| Juice .. | 18·40 | 67·2 | —18·7 | 67·1 | —0·1 | 1·46 | —2·0 | 16·22 | 1·01 | 0·45 |
| Molasses | 72·3 | 51·2 | —14·8 | 51·6 | —0·4 | 16·30 | —2·45 | 51·6 | 10·1 | 6·2 |
| Juice .. | — | 69·4 | —20·0 | 69·8 | —0·4 | 1·70 | —6·7 | 16·86 | 1·02 | 0·68 |
| Molasses | 73·6 | 52·3 | —15·6 | 53·0 | —0·7 | 15·30 | —4·5 | 53·0 | 9·3 | 6·0 |
| Juice .. | 19·7 | 70·5 | —20·0 | 70·7 | —0·2 | 1·16 | —1·4 | 16·92 | 0·81 | 0·35 |
| Molasses | 70·9 | 49·8 | —14·8 | 50·5 | —0·7 | 13·20 | —5·3 | 70·9 | 8·0 | 5·2 |
| Juice .. | 19·2 | 67·5 | —19·25 | 67·7 | —0·2 | 1·61 | —3·6 | 16·21 | 1·06 | 0·56 |
| Molasses | 74·5 | 50·5 | —15·1 | 51·2 | —0·7 | 15·6 | —4·5 | 74·5 | 9·5 | 6·1 |
| Juice .. | 16·0 | 48·5 | —13·9 | 48·7 | —0·2 | 2·39 | —2·0 | 11·88 | 1·59 | 0·80 |
| Molasses | 76·6 | 48·6 | —13·5 | 48·5 | —0·1 | 17·1 | —0·6 | 76·6 | 10·3 | 6·8 |
| Juice .. | 18·8 | 61·0 | —17·8 | 61·6 | —0·6 | 2·0 | —7·1 | 14·88 | 1·30 | 0·70 |
| Molasses | 77·8 | 49·4 | —15·0 | 50·3 | —0·9 | 14·7 | —6·1 | 77·8 | 8·8 | 5·9 |
| Juice .. | 19·1 | 69·1 | —18·9 | 68·8 | —0·3 | 1·42 | —5·4 | 16·56 | 0·92 | 0·50 |
| Molasses | 73·0 | 51·0 | —15·1 | 51·6 | —0·6 | 13·7 | —4·4 | 73·0 | 8·5 | 5·2 |
| Juice .. | 19·3 | 68·4 | —16·2 | 69·2 | —0·8 | 2·27 | —9·1 | 16·66 | 1·45 | 0·82 |
| Molasses | 77·0 | 46·4 | —14·8 | 47·8 | —0·4 | 20·0 | —7·0 | 77·0 | 12·0 | 8·0 |
| Juice .. | 19·0 | 70·0 | —20·0 | 70·3 | —0·3 | 1·22 | —6·4 | 16·92 | 0·79 | 0·43 |
| Molasses | 76·0 | 45·0 | —13·4 | 45·7 | —0·7 | 20·7 | —3·4 | 76·0 | 12·8 | 7·9 |
| Juice .. | 17·9 | 64·5 | —18·5 | 65·0 | —0·5 | 1·46 | —8·6 | 15·62 | 0·92 | 0·54 |
| Molasses | 74·0 | 41·3 | —13·9 | 43·1 | —1·8 | 22·8 | —7·9 | 74·0 | 13·7 | 9·1 |

The average rotatory power of the reducing sugars in 67 samples of exhausted molasses was −20·5, which indicates considerable inversion during the later stages of manufacture.

**Formation of Organic Acids from Reducing Sugar.**

It is easy to ascertain the amount of organic acid formed by the action of lime on reducing sugar by determining the percentages of lime and carbonic acid in the ash of unclarified and clarified juice. In the analyses given below the amount of lime combined with phosphoric acid is not taken into account, as this is removed from the juice during defecation. In the raw juice, the content of lime not combined with phosphoric acid, the free acid, and the carbonic acid in the ash are given; and in the clarified juice, the lime not combined with phosphoric acid, and the carbonic acid in the ash. The free organic acid combines with lime during defecation and yields (even without decomposition of the reducing sugar) more

lime salt in the juice and carbonic acid in the ash. If an excess of these latter is found in the clarified juice this is owing to the formation of lime salts by decomposition of reducing sugars, and may serve as a measure for same.

In order to settle this point the author determined the free acid and lime in the raw juice on four Java estates, and the lime-content of the same juice after clarification and subsiding, namely that portion of the lime not combined with phosphoric acid, and which was not precipitated by ammonia from a dilute hydrochloric acid solution of the ash previously freed from silica.

| Constituents. | I. | | II. | | III. | | IV. | |
|---|---|---|---|---|---|---|---|---|
| | Raw Juice. | Clarified Juice. | Raw Juice. | Clarified Juice. | Raw Juice. | Clarified Juice. | Raw Juice. | Clarified Juice. |
| Brix .. .. .. .. .. | 16·3 | 17·6 | 15·7 | 17·9 | 15·9 | 15·9 | 17·2 | 19·0 |
| Free acid as $H_2SO_4$ ... | 0·034 | — | 0·037 | — | 0·043 | — | 0·038 | — |
| Ash .. .. .. .. .. | 0·574 | 0·146 | 0·358 | 0·149 | 0·352 | 0·318 | 0·231 | 0·179 |
| Lime not combined with phosphoric acid .. .. | 0 | 0·016 | 0·004 | 0·020 | 0 | 0·028 | 0·002 | 0·023 |
| Increase of lime .. .... | — | 0·016 | — | 0·016 | — | 0·028 | — | 0·021 |
| Equivalent with $H_2SO_4$ .. | — | 0·030 | — | 0·030 | — | 0·049 | — | 0·037 |
| Tempered with litres lime cream of 15°B. on 1000 L. of juice .. .. .. .. | 4½ | — | 3 | — | 4½ | — | 4½ | — |

We see that the increase in the percentage of lime present as a soluble lime salt is almost equivalent to that of the free acid in the raw juice, so that this increase is only due to neutralization of free organic acid and not to formation of lime salts of organic acids arising from the decomposition of reducing sugars.

**Gummy Matter and Pectine.**

Gum is not precipitated by lime in slightly alkaline liquids, for a solution containing 15 per cent. of sucrose and 0·121 per cent. of cane gum, after ordinary defecation, still contained 0·120 per cent. of gummy matter, *i.e.*, the original amount. This percentage did not fall after liming, heating, and treating with sulphurous or phosphoric acids, &c., showing that defecation does not remove the cane gum which is extracted from the cane fibre under heavy pressure.

The dissolved pectin is to some extent precipitated during defecation. A juice which contained 0·634 per cent. of total gums and pectin in the raw state, contained only 0·342 per cent. of these after defecation. Maxwell* found that a large quantity of gum was separated by liming cold, while Winter† mentions a slimy precipitate formed in limed juice and which proved to consist of a compound of lime and a carbohydrate. It contained galactose compounds, but as this substance differs much from galactan, it was more probably a lime salt of pectin.

* *Report of the Hawaiian Exp. Station*, 1896, 54. † *Uit het chemische Laboratorium*, 49.

Xylan was not present, otherwise it would have been recognised among the products of hydrolization, by the melting point of the osazones, but this was not the case.

Maxwell* ascertained the percentage of nitrogenous matter (amid and albuminoid nitrogen) in juice during the different stages of manufacture.

**Nitrogenous Bodies.**

Clarified juice contained 2·1 parts of nitrogenous matter and syrup 2 parts on 100 parts of dry substance so that a clarified juice of 16 Brix contained 0·32 per cent. of nitrogenous bodies or about 0·05 per cent. of nitrogen.

Van Lookeren Campagne† found much lower percentages of amids and other nitrogenous substances, varying between 0·044 and 0 082 per cent. in undiluted juice and in diluted mixed juice between 0·070 and 0·016 per cent.; so that the nitrogen calculated from these data does not exceed 0·02 per cent.

According to Maxwell, in 100 parts of total nitrogen there are :—

| | Nitrogen as Albuminoid. | Nitrogen as Amid. |
|---|---|---|
| Mill juice .. .. .. .. .. .. .. | 35 | 65 |
| Diffusion juice (cold) .. .. .. .... | 24·5 | 75·5 |
| Diffusion juice (hot).. .. .. .. .. | 10·9 | 89·1 |
| Clarified juice.. .. .. .. .. .... | 3·3 | 96·7 |
| Syrup .. .. .. .. .. .. .. .. | 3·2 | 96·7 |

And according to van Lookeren Campagne :—

| Description of the Juice. | Total Nitrogen. | Nitrogen × 6·25. | Albuminoids. | Amids and similar bodies. | Albuminoids on 100 nitrogenous matter. |
|---|---|---|---|---|---|
| Diluted mixed juice (factory mill).. .. | 0·024 | 0·150 | 0·057 | 0·093 | 38 |
| ,, ,, ,, .... | 0·024 | 0·150 | 0·063 | 0·087 | 42 |
| ,, ,, ,, .. .. | 0·028 | 0·175 | 0·059 | 0·116 | 34 |
| Undiluted cane juice (test mill) .. .... | 0·021 | 0·131 | 0·061 | 0·070 | 47 |
| ,, ,, ,, .. .. | 0·026 | 0·162 | 0·080 | 0·082 | 49 |
| ,, ,, ,, .. .... | 0·015 | 0·097 | 0·053 | 0·044 | 55 |
| ,, ,, ,, .. .. | 0·020 | 0·125 | 0·058 | 0·068 | 46 |

* *Bull. Assoc. Chim. Sucr. et Dist.*, 1895, 371.

† *Archief voor de Java Suikerindustrie.*

The diluted juices were not extracted from the same canes as the undiluted juices; the latter being extracted by a laboratory mill and therefore contained less albuminoids (*vide* page 121).

A juice containing 0·057 per cent. of albuminoids lost 0·047 per cent. or 82 per cent. of the total on defecation, and another juice lost 0·067 out of 0·080 or 84 per cent. of the total, so that 17 per cent. passes over into the clarified juice.

## CARBONATATION.

### A.—Separation of the Juice.

Juice treated by the defecation process still contains so many gummy constituents that it is very difficult to filter, and is therefore never quite clear.

**Principle.** The manufacture of white granulated sugar demands more care and necessitates filtrations of the clarified juice as well as of the syrup, and consequently a more thorough elimination of impurities than the simple defecation process permits of. Such purification is brought about by adding a large excess of lime, which precipitates albumen and gums and decomposes reducing sugars. We have already mentioned that when this action of lime takes place at the temperature of boiling juice, the lime salts are dark-coloured, viscous, and very troublesome, but if the lime acts upon glucose at temperatures below 55°, the lime salts are partly insoluble, and easily removed, the remainder being colourless and very soluble. As soon as the lime has finished its purifying action, the excess is removed by carbonic acid, which combines with the lime and forms insoluble carbonate of calcium. This saturation may be effected in one or in two operations, and according as this is done the process may be distinguished by the name of single or double carbonatation, both of which have their advantages as we shall show later on.

**Description of the Carbonatation Process.** The mill juice is mixed with as much lime-cream as is thought necessary in large tanks provided with stirring apparatus, and then passes through juice heaters, where it is warmed to 40-50° C. From the heaters it enters the carbonatation tanks, which are only half filled with the juice so as to allow for the violent frothing which occurs during saturation with the gas. Sometimes these tanks are open, but in most cases they are covered with an iron plate provided with a lid, permitting samples of the contents to be taken from time to time, and allowing entrance to the tank for cleaning it. A chimney carries off the escaping gases. A coil, into which steam can be admitted, serves for heating the juice, and a perforated iron coil or cross for introducing the carbonic acid.

As soon as the tank is half filled with the heated and limed juice, carbonic acid is turned on, care being taken that the temperature shall not exceed 55° C.

At first the carbonic acid is completely absorbed, but the juice soon becomes thick and froths violently, and may even overflow the tank if the lid in the cover is not kept closed. Indeed the froth sometimes rises into the chimney, causing the inner surface of the latter to be coated with juice and calcareous mud. In order to moderate the foaming a little oil is poured on the froth, or it is blown down by a jet of steam, though in the latter case one must be very careful not to overheat the alkaline juice locally and thus give rise to dark-coloured products in the juice.

**Frothing of the Juice during Saturation.**

Whilst the juice remains gelatinous the absorption of carbonic acid is very incomplete, and the gases escaping up the chimney still contain much unabsorbed and therefore wasted carbonic acid. Later on, when the juice becomes more liquid, the carbonic acid is absorbed more easily, and a moment arrives when the precipitate settles rapidly, and a sample taken from the tank separates into a bright coloured alkaline liquid, and a greyish-yellow, rapidly subsiding precipitate. Up to this point the procedure is similar in both single and double carbonatations, but this is so no longer.

**Absorption of the Gas.**

In the single carbonatation the carbonic acid is admitted until a sample of the juice gives a faint pink reaction on phenolphthalein paper, the supply being then slowly shut off, the juice being just neutral. It is afterwards heated by the steam coil up to 90° C., and filtered through filter-presses, or if the heating of juice containing such a large quantity of scum should be difficult, the saturated juice may be run off into settling tanks. The clear juice (which is obtained immediately) is syphoned off, the scum steamed with direct steam, and then filtered. The quantity of lime necessary for this operation is entirely regulated by the running of the filter-presses; the precipitate from the juice is rather viscous, and is very difficult to filter, therefore carbonatation aims at mixing it with so much carbonate of lime that the mixture can be easily filtered. If the filter presses run slowly before the chambers are full of scum, the quantity of lime added to the juice should be increased, and as an excess of lime is perfectly harmless in this process we cannot use too much, because the carbonic acid is certain to neutralize it. We may, therefore, go on for a long time with one fixed quantity of lime and not have to put up with the inconvenience met with in the defecation process, occasioned by the difference in quality of the juice of canes from various fields.

**Single Carbonatation.**

If, on the contrary, the filter-presses work rapidly, the proportion of lime can be reduced without harm ensuing; but generally the same proportion of lime cream is maintained throughout the whole grinding season. As a rule the single carbonatation requires 1 per cent. of lime on 100 cane, or 70 litres of lime cream at 15° Bé. on 1000 litres of juice.

**Double Carbonatation.**

In the double carbonatation process the supply of carbonic acid is stopped as soon as the juice settles rapidly; it then still possesses an alkalinity of 0·05 per cent. CaO. A cock is opened at the bottom of

the saturation tank and the whole contents are filtered through presses without heating; and therefore at a temperature of under 60° C.; great care should be taken to stop this first saturation at the exact point when the precipitate settles well, while the juice is still alkaline. If stopped too early the juice does not filter well, because it is still too thick and the precipitate may contain undecomposed lime saccharate. If it is allowed to go too far the juice becomes neutral again and is able to redissolve the impurities already precipitated, and which are insoluble in alkaline juice, but now redissolve and impart a brown colour to the liquid.

**First Carbonatation.**

The filtered alkaline juice is pumped into other saturation tanks, which are also fitted with coils and pipes for steam and carbonic acid, but which may be filled to the brim, as in these the juice no longer froths. A slight quantity of lime is added, and saturation repeated up to neutrality; then the juice is boiled to break up bi-carbonates, and again filtered through filter-presses.

**Second Carbonatation.**

Just as with single carbonatation, in this case also the quantity of lime is regulated by the filter-presses. As the juice is now filtered at a low temperature, and in that case the precipitate from the juice is still more difficult to deal with than at a more elevated one, a still greater quantity of lime is required to obtain good hard filter-press cakes, and hence double carbonatation requires more lime and also more carbonic acid than the single process.

**Determination of the Amount of Lime-Cream.**

When the slow filtration indicates that the precipitate is too viscous, the quantity of lime must be increased, and in this case also a maximum may be used, because the excess is neutralized by the acid. As a rule we go as far as 1½ per cent. of the weight of cane, or about 100 litres of lime-cream of 15° Beaumé to 1000 litres of juice.

Sometimes the juice of canes from certain fields, especially those having a soil rich in humus or being heavily manured, or juice from fallen or from immature canes, contains such an enormous amount of albuminoid matter that it becomes too thick and froths so violently that the saturation is seriously interfered with.

The great bulk of the carbonic acid passes unutilized in large bubbles through the liquid, which prolongs the saturation time to twice or thrice its original duration. Little can be done to prevent this excessive foaming, though theoretically it might be combatted by an increased addition of lime, but then the cost becomes too great, and the saturation of the excess of lime requires extra time, while finally the larger amount of filter-press cakes involves a larger loss of sucrose contained in them. But if one has a field of cane yielding juice which requires more than two per

**Difficulties met with in Saturation.**

cent. of lime (on the weight of cane), such juice should not be worked up alone, but mixed with juice of a more normal character.

Sometimes the sweet waters of the filter presses are utilized for the preparation of the lime cream, but this is a practice to be strongly condemned, as it often happens that for some unknown reason such lime cream produces very viscous and sticky juice which is difficult to saturate, but which again becomes tractable as soon as pure water is used for slaking the lime.

**Third Saturation with Sulphurous Acid.**

In many cases it is impossible to obtain quite neutral juices by saturation with carbonic acid, as either the lime or the juice contains alkali, which becomes carbonate and thus keeps the juice alkaline after saturation. In order to prevent this, and also with a view to obtaining a still further decoloration, a third saturation, this time with sulphurous acid, is sometimes used in factories working with the double carbonatation process. To this end the saturated juice is pumped into elimination pans, and sulphurous acid gas let in, until the juice is neutral to phenolphthalein paper, and remains so after being boiled, after which it is filtered. Great care is advisable in this operation, for an excess of sulphurous acid is very injurious, as will be shown in the Section on Sulphurous Acid.

## B.—Influence of Carbonatation on the Constituents of Cane Juice.

We have already seen that the chief object of the carbonatation process is to obtain an easily filtered juice, and that to this end, an amount of lime was necessary which was disproportionately large in relation to its real purifying action, and which constitutes (as calcium carbonate) the bulk of filter press cakes.

**Reducing Sugars necessitate Working at Low Temperatnre with additional Lime.**

In addition to the impurities precipitated by simple defecation, carbonatation (especially double carbonatation) removes part of the reducing sugars in the form of organic lime-salts. It must be observed that this is not the object, but only a secondary feature of the carbonatation, and one which we have learned to minimize by keeping the temperature low during the saturation, but which is more costly, as it involves the use of more lime and coke, and also occupies more time.

**Composition of the Filter Press Cakes.**

The analyses of the filter press cakes show that they chiefly consist of precipitated calcium carbonate with only small percentages of substances derived from the juice, among which bagasse fibre, wax and albumen are the most prominent.

The composition of some samples is recorded here, calculated on the dry substance; it may be added that in a fresh state they contained about 50 per cent of moisture more or less.

| Constituents. | Filter-press Cakes from Single Carbonatation. | Filter-press Cakes from Double Carbonatation. 1st Saturation. | 2nd Saturation. |
|---|---|---|---|
| Sucrose | 3·90 | 10·10 | 8·10 |
| Glucose | — | 0·38 | — |
| Cane fibre | 3·08 | 6·10 | 0·50 |
| Wax | 3·43 | 2·48 | — |
| Organic Acids | 1·94 | 2·12 | 4·20 |
| Albumen | 2·55 | 2·72 | — |
| Gums | 1·11 | 1·20 | — |
| Total organic substances | 16·01 | 25·10 | 12·80 |
| Calcium carbonate | 67·94 | 61·94 | 85·01 |
| Magnesium carbonate | 1·80 | 1·21 | 0·10 |
| Iron and Alumina | 2·41 | 4·66 | 0·51 |
| Phosphoric acid | 0·71 | 0·87 | 0·12 |
| Silica | 8·48 | 1·85 | 0·64 |
| Sand and clay | 2·48 | 4·37 | — |
| Undetermined inorganic substances | 0·17 | — | 0·82 |
| Total inorganic substances | 83·99 | 74·90 | 87·20 |

**Amount of Impurities from the Juice removed by Carbonatation.**

The large amount of inorganic matter in the press cake renders it difficult to calculate the amount of solid matter separated from the juice during carbonatation. It must, however, be greater than that removed during simple defecation as indicated by the quotient of purity, though it is true that a part of that increase is only apparent, as we shall see later on.

**Comparison of the Purifying Action of Carbonatation and Defecation.**

The purifying action of carbonatation may be judged from the following analyses of cane juice, one portion of which was clarified by the defecation process and the other by carbonatation. This latter is somewhat diluted by the water added in the form of lime-cream.

| | Brix. | Sucrose. | Reducing Sugar. | Pectin. | Glucose Ratio. | Quotient of Purity. |
|---|---|---|---|---|---|---|
| Original mill juice | 18·4 | 15·72 | 0·92 | 0·64 | 5·9 | 85·6 |
| Clarified by defecation | 18·6 | 15·99 | 0·95 | 0·29 | 5·9 | 86·1 |
| Clarified by carbonatation | 16·9 | 14·65 | 0·44 | 0·14 | 3·0 | 86·7 |

The effect of carbonatation on the constituents of the juice is as follows:—

At the low temperature prevailing during carbonatation lime does not act destructively on sucrose and since the time during which the juice is heated is very short, we may neglect such trifling losses of sucrose. There exists, however, another source of loss, viz., that as calcium saccharate in the filter-press cake. We mentioned that during the course of saturation the juice becomes thick and viscid and, when filtered at that stage and analysed, we find a rather considerable loss of sucrose, as compared with the original juice. Evidently a part of the sucrose has been withdrawn from the liquid in an insoluble form, namely, as calcium hydro-carbonate and saccharate. When the juice is completely saturated, this combination becomes broken up into calcium carbonate and free sucrose, so that the precipitate no longer retains sucrose in an insoluble form in the precipitate.

**Effect of Carbonatation on the Constituents of the Juice.**

**Loss of Sucrose as Calcium Saccharate.**

But if saturation is stopped before it is completely finished or if the lime has been too coarse and contained unsaturated lumps, which, by their gradual dissolution after the end of the saturation augment the content of dissolved lime, there is danger of insoluble calcium saccharate remaining in the filter-press cakes. This cannot be decomposed by steaming or washing, nor can it be detected by the usual analysis, but only if ammonium nitrate be added to the water with which the filter-press cake is triturated for analysis. This reagent combines with the lime, and liberates the sucrose.

The extent of the loss of sucrose in such a case cannot be expressed in figures; it is usually *nil*, but no one can tell how large it may become if the work is performed carelessly. Weisberg* mixed a solution of white sugar of 14° Brix with lime cream to an alkalinity of 2·3 per cent. CaO. This solution after total saturation was found to contain 12·89 per cent. of sucrose. The mixture was saturated with carbonic acid until a gelatinous precipitate formed, which was filtered off (a troublesome operation), but the filtrate became turbid again on exposure to the atmosphere. The alkalinity then amounted to 1·95 per cent., and after neutralization the filtrate showed a sucrose content of 12·71 per cent. Prolonged saturation produced an abundant white gelatinous precipitate, and the liquid became still more viscous. A part of it was filtered with much trouble, but the filtrate soon became turbid, and still showed an alkalinity of 0·52, but the sucrose had been reduced to 11·80 per cent. After complete saturation a clean and easily filtered liquid was finally obtained, which gave the same polarization as the original liquid, so that ultimately all the precipitated sucrose had gone into solution again. Consequently, at the moment of maximum viscosity, no less than 1·09 out of 12·09 of sucrose, or 8·5 parts per cent., were precipitated, and would have been lost if the liquid had been filtered at that

**Importance of the loss of Sucrose as Calcium Saccharate.**

* *Bull. Assoc. Chim. Sucr. et Dist.*, 1898-99, 177.

stage. When filtering at a later stage, but before the combination is entirely broken up, the loss is of course less, but still remains a loss. We cannot, therefore, give exact figures, but would impress on chemists the great necessity of exercising thorough control over the filter presses and the termination of the saturation, *i.e.*, the point at which the juice settles most rapidly; further, treatment with ammonium nitrate of that portion of the filter-press cakes, which is employed for the determination of its sucrose content.

**Reducing Sugar.** The effect of carbonatation on reducing sugar is twofold, viz., destructive and transforming.

**Destructive Action of the Lime.** Reducing sugar is broken up by lime into organic acids which immediately combine with that base in proportions depending on the temperature, the concentration both of lime and of sugar, and the duration of contact. We have repeatedly stated that this action takes place very rapidly at high temperature, and that under these circumstances a very complete decomposition of the sugar-molecules results, with the formation of gaseous and humic products. At low temperatures, however, the sugar merely undergoes an intermolecular re-arrangement, rather than decomposition, as the principal bodies formed, lactic acid ($C_3H_6O_3$) and saccharic acid ($C_6H_{12}O_6$), have the same elementary composition as the sugar from which they arise, viz., $C_6H_{12}O_6$.

**Spontaneous Decomposition of Derivatives of Reducing Sugar.** As long as the temperature remains under 55° during carbonatation, we chiefly notice the last mentioned reaction, but if by some accident or carelessness the temperature of the alkaline cane juice rises, very unstable acids are formed which remain intact as long as they occur in weak concentration, but become concentrated in the molasses (especially if the latter are re-boiled). These may undergo spontaneous decomposition; and the whole mass has sometimes been observed to foam up with considerable rise of temperature, and finally change into a black, carbonaceous and porous mass, having a very acid reaction due to formic acid. Even if the decomposition does not go as far as this, so much formic acid is formed that the molasses loses much sucrose by inversion. This is the reason why the first requirement in carbonating cane juice is to keep the temperature down to 55°C. until complete neutrality is obtained by saturation. Assuming that this rule is well understood and carried out, we may confine our attention to the effects of lime on the reducing sugar at that temperature.

**Action of Lime at Temperatures under 55°C.** A solution of 16·39 per cent. sucrose and 2·07 per cent. glucose was mixed at 55°C. with a 10 per cent. (by volume) lime cream at 20° Bé. and kept at that temperature for one hour. From time to time a sample was withdrawn from the mixture and analysed with these results.

| | Sucrose. | Glucose. | Glucose Ratio. |
|---|---|---|---|
| Original solution .. .. .. .. .. | 16·39 | 2·07 | 12·— |
| After 5 minutes .. .. .. .... | 13·39 | 1·38 | 10·3 |
| ,, 10 ,, .. .. .. .. .. | 13·36 | 1·22 | 9·1 |
| ,, 15 ,, .. .. .. .... | 13·40 | 1·05 | 8·— |
| ,, 22½ ,, .. .. .. .. .. | 13·39 | 0·75 | 5·6 |
| ,, 30 ,, .. .. .. .... | 13·40 | 0·65 | 4·9 |
| ,, 45 ,, .. .. .. .. .. | 13·40 | 0·55 | 4·1 |
| ,, 60 ,, .. .. .. .... | 13·38 | 0·45 | 3·3 |

The glucose diminished steadily, being reduced to half its original amount within 20 minutes, but was not completely decomposed after an hour's standing. At the end of the 60 minutes, the liquid, which at the outset had been free from lime salts, contained (in addition to a large excess of uncombined lime) 0·328 per cent. CaO combined with organic acids. A part of the glucose was therefore still present in the solution but changed into organic acid, and that as a salt or mixture of salts, having a lime content of 21·1 per cent. CaO. The liquid had assumed a light-yellow tint during the operation. Several portions of a solution, containing 15·35 per cent. sucrose and 2 per cent. glucose were next mixed with different amounts of lime cream kept at 55° C. for a quarter of an hour and then analysed.

The dilution was different in each sample and the figures are, therefore, not mutually comparable, except those of the glucose ratio which are all of them calculated to the basis of 100 sucrose.

| | Sucrose. | Glucose. | Glucose ratio. | Alkalinity in % CaO. |
|---|---|---|---|---|
| Original solution .. .. .. .. | 15·35 | 2·— | 13·— | — |
| With 1% lime cream .. .. .... | 14·85 | 1·83 | 12·3 | 0·142 |
| ,, 2% ,, .. .. .. | 14·66 | 1·70 | 11·6 | 0·280 |
| ,, 3% ,, .. .. .... | 14·50 | 1·59 | 11·0 | 0·403 |
| ,, 5% ,, .. .. .. | 14·10 | 1·47 | 10·4 | 0·583 |
| ,, 7% ,, .. .. .... | 13·90 | 1·22 | 8·7 | 0·740 |
| ,, 10% ,, .. .. .. | 13·51 | 0·92 | 6·8 | 1·210 |

This shows that the reducing sugar, in this case glucose, disappears as such under the action of the lime, and the question arises whether the products of its decomposition remain dissolved in the juice or are precipitated and removed in the filter press cakes, and whether there is any difference in this respect between single and double carbonatation; in other words, whether these products of decomposition are more or less soluble in alkaline than in neutral sugar juices.

**Tracing the Products of Decomposition.**

A neutral solution of 18·41 per cent. sucrose and 1·76 per cent. glucose was mixed with 10 per cent. of lime cream (by volume) and kept at 55° C. for a quarter of an hour, then saturated with pure carbonic acid until the precipitate, which was first gelatinous, settled well. The liquid, being still alkaline, (0·056 per cent. CaO) was then divided into two parts. The colour of the liquid was bright yellow, that of the precipitate a dirty yellow. One of the two parts was saturated until a drop of the solution placed on a piece of phenolphthalein paper only gave a faint pink reaction. The liquid was boiled, filtered and represented the juice clarified by the single carbonatation process. The other part was filtered at 55° C., a few drops of lime milk added to the bright-yellow filtrate, which was then saturated with carbonic acid until it gave a neutral reaction, heated, filtered, and represented cane juice clarified by the double carbonatation process. The essential difference, therefore, consists in the first precipitate being produced in a neutral medium, whereas the second is removed when the liquid is still alkaline, so that re-solution of the precipitate is avoided. The analyses of the juices gave the following results :—

| | Sucrose. | Glucose. | Glucose Ratio. | CaO. | Colour |
|---|---|---|---|---|---|
| Original solution .. .. .. | 18·41 | 1·76 | 9·1 | 0· | Colourless. |
| Saturated to 0·056 per cent. CaO. | 16·48 | 0·96 | 5·8 | 0·158 | Light yellow. |
| Single carbonatation.. .. .. | 16·50 | 1·05 | 6·3 | 0·148 | Yellow. |
| Double carbonatation .. .... | 16·50 | 0·96 | 5·8 | 0·127 | Light yellow. |

The solution clarified by single carbonation had a yellow colour, a glucose ratio of 6·3, and contained 0·148 per cent. CaO combined with organic acids, while the liquid which was saturated by double carbonatation (after removal of the precipitate produced in the alkaline medium) had a lighter colour, a glucose ratio of 5·8, and only contained 0·127 per cent. CaO as organic lime salts. The prolonged saturation in the first instance had therefore redissolved reducing matter as well as lime salt and colouring matter from the precipitate obtained at the first saturation. We can prove by direct experiment that this precipitate does contain these substances by suspending it in water and decomposing it with carbonic acid. On filtering the liquid, we obtained a coloured filtrate, which contained lime salts and reduced Fehling's test solution.

The cuprous oxide precipitated from the test solution by the filtrate is not bright-red as usual but greenish-yellow, a proof that the reducing body is not a reducing sugar, but one of its products of decomposition having a different reducing power. But apart from chemical analysis we can judge from the appearance of the precipitate that something has happened during complete

saturation, as it suddenly changes from a yellow, voluminous precipitate into a greyish, granular one, as soon as the point of complete neutralization is reached.

Carbonatation of cane juice produced similar results.

| | Brix. | Sucrose. | Reducing Sugar. | Purity. | Glucose Ratio. | Combined CaO. |
|---|---|---|---|---|---|---|
| Cane juice .. .. .. .. | 17·7 | 16·09 | 0·55 | 90·9 | 3·4 | — |
| Juice with lime at 55°C.... | — | 14·47 | 0·30 | — | 2·— | — |
| Saturated to 0·040% CaO. .. | 15·8 | 14·93 | 0·29 | 94·5 | 2·— | 0·036 |
| Single carbonatation .... | 16·— | 14·93 | 0·36 | 93·3 | 2·4 | 0·056 |
| Double carbonatation.. .. (Diluted during second saturation) | 15·3 | 14·50 | 0·17 | 94·6 | 1·2 | 0·029 |

In the first place, the lime salt of an organic acid, formed from the reducing sugar, is precipitated and afterwards redissolved on neutralizing. Consequently, double carbonatation not only attacks a portion of the reducing sugar but causes a partial removal of same in the filter-presses, while single carbonatation, which also attacks a part of the reducing sugar, leaves the products of decomposition in the juice, so that this is more strongly coloured and contains more lime salts than the one clarified by double carbonatation.

The analysis of the filter-press cake also gives evidence of the fact that double carbonatation precipitates more organic lime salts than the single operation. A quantity of the cake obtained after the first saturation under 55° C. and filtered at that temperature, was washed with cold water and this liquid evaporated. It then showed the same composition as the juice from which the cakes had been precipitated, thus proving that cold water does not extract matters previously precipitated from it. 100 parts of dry substance in this solution contained no more than 0·55 parts of CaO. The extracted cake was next boiled with water and yielded a solution containing no less than 18 parts of CaO on 100 parts of dry substance. When the cake was suspended in water and treated with a current of carbonic acid, much more organic matter became dissolved and the liquid assumed a brown colour.

**Amount of Precipitated Lime Salts.** The lime salts mentioned here are only a part of the total quantity of those formed from the reducing sugar; in this instance 0·021 out of 0·148 or 14 per cent., and 0·027 out of 0·056 or 48 per cent.; the balance is formed by calcium lactate and saccharinate, both of them being soluble and stable salts, and not liable to spontaneous decomposition.

**Total Amount of Lime Salt.** The total amount of lime salt arising from the action of lime on reducing sugar varies greatly, and no exact figures can therefore be given. High temperatures yield more than low ones, as is shown from the instances cited below, where the figures represent per cent. CaO in the carbonated juices.

| | Per cent. |
|---|---|
| Double carbonatation at 40°C. .. .. .. .. .. | 0·0144 |
| ,, ,, 40°C. .. .. .. .. .... | 0·021 |
| ,, ,, above 60°C. .. .. .. .. .. | 0·795 |
| Single carbonatation at 50°C. .. .. .. .. .... | 0·0514 |

**Effect of Lime on Optical Activity.**

The mixture of reducing sugars is rendered optically inactive by the action of lime and, therefore, its initial levo-rotation decreases, and the dextro-rotation of the total mixture of sugars is slightly increased as also the quotient of purity, so that the increase in purity is partly due to clarification and partly to changes in the rotatory power of one of the constituents of the juice. Taking the specific rotatory power of the reducing sugar as—6° Ventzke and its percentage as 1 per cent., we see that the levo-rotation of the reducing sugar neutralizes the dextro-rotation of 0·06 per cent. sucrose. Now this levo-rotation is destroyed by the action of the lime, so that an apparent increase in the sucrose of 0·06 per cent. is obtained or an increase in the purity of 0·5. This figure is so insignificant that in reality the increase of purity observed during carbonatation is chiefly due to the precipitation of dissolved impurities.

**Gummy Matter.**

Carbonatation precipitates much more of the gummy matters than simple defecation. A solution containing 15 per cent. of sucrose and 0·121 per cent. of cane gum was treated with lime and carbonic acid in the same manner as juice, and saturated after the single and the double carbonatation process. After treatment it contained respectively only 0·036 per cent. and 0·040 per cent. of cane gum, showing that about 70 per cent. of this body was removed.

**Lime Substitutes.**

We have already observed in the previous pages that with carbonatation much more lime was applied than contributed to the clarifying action, and for the sole reason that otherwise the filtration would be difficult or even impossible. As lime is a rather expensive material and as its transformation into calcium carbonate requires time and carbonic acid, it is not surprising that various attempts have been made to economize lime and carbonic acid by substituting other filtering media for the calcium carbonate formed during saturation. Dabrowski and Kaczmarkiewicz recommended the addition to the juice of 1 per cent. by weight of powdered calcium carbonate in the form of limestone, chalk or marl, and reducing by one half the usual amount of quick lime Other experimenters suggested 2 per cent. of kieselguhr or infusorial earth in place of calcium carbonate and half the usual dose of lime. Kuthe and Anders mixed the juice with filter-press cakes from a former operation and also half the usual addition of lime by stirring the lime cream with filter-press mud and using this mixture for liming the juice. Though, from time to time, these and similar expedients have been recommended and occasionally find enthusiastic advocates, they have not as yet come into regular practice, and have always been speedily abandoned wherever they were tried.

A better result is obained by the substitution for a part of the calcium carbonate of the more easily filtered sulphite. When, along with the carbonic acid, a little sulphurous acid is applied during saturation, not only does this operation proceed much more rapidly, but harder and dryer press-cakes are obtained even with less lime. The supply of sulphurous acid is turned off together with that of the carbonic acid as soon as the precipitate settles well, and the liquid is therefore still alkaline, so that no danger can exist of the carbonate being converted into soluble bisulphite.

**Simultaneous Saturation with Carbonic and Sulphurous Acids.**

Finally, we may consider the combination of defecation with carbonation in such a manner that the bulk of gummy impurities is first removed by defecation, the cooled juice being afterwards mixed with an excess of lime and saturated with carbonic acid. The gummy constituents, which are difficult to filter, being already removed, the resulting defecated juice does not require so much lime to yield an easily filtered juice as when the raw juice had been submitted to carbonatation without previous defecation. This method has the advantage of saving lime, but a great deal of this saving is lost again by the expense of heating the juice twice, first on defecation and again after the second saturation. A more serious drawback is that there is always a danger of the defecated juice entering the carbonatation tanks at too high a temperature owing to insufficient cooling, and even the most careful supervision fails to prevent an occasional tankful of juice being limed at too high a temperature with all the undesirable consequences resulting therefrom.

**Combination of the Defecation and Carbonatation Processes.**

### C.—Advantages and Defects of Single and Double Carbonatation.

The advantages of carbonatation over defecation are that the juice is better purified and freed from gummy constituents, is easier to filter, and yields a more fluid molasses. Yet, when we lime the juice cold and, before heating, remove the excess of lime by sulphurous acid, a great improvement in this direction results. Another considerable advantage is that the clarification is effected in a single operation, for, immediately after saturation, the liquid is divided into a clear juice, easy to filter, and a precipitate which settles rapidly; whereas, in the case of defecation, with or without sulphurous acid, the precipitate settles slowly and filters badly, whilst the clarified juice is gummy and opalescent.

Independent of the kind of cane crushed, we can employ a like quantity of lime for a long time at a stretch, because if an excess of lime has been used, the carbonic acid neutralizes it afterwards, while the quantity of lime required in defecation is very variable if canes from two different fields are crushed together, and therefore one is not sure of using it in proper quantity, which is a very important point in this operation.

Owing to the carbonated juice being purer, brighter, and more easily worked, the quality of the sugar is better than from similar juice clarified by the defecation process.

Against these advantages must be set certain drawbacks, the chief of which is the greater expense, because more lime is required; and the cost of the lime-kiln and fuel, together with that of the carbonic acid pump and other plant, which increases the cost price of the sugar.

Further, the work connected with the lime-kiln requires continuous attention; it often occurs that either the lime is burnt at too high or two low a temperature, or that the quality of the gas is deficient, and there are other drawbacks that do not occur in the defecation process.

If we do not expect more from the carbonatation process than a more perfect subsidation and clarification of impure juices and do not want to make white sugar, the single carbonatation process is amply sufficient. But even then it is too expensive because we can obtain this by liming the cold juice heavily and saturating it still cold with sulphurous acid, which is a very convenient and inexpensive process.

If, on the other hand, we want to make sugar for direct consumption, then everything apt to cause coloration should be avoided, and we must have recourse to the double carbonatation process. This requires much lime and carbonic acid, and thus causes considerable expense, but this is more than compensated for by the higher price which such sugar fetches when compared with raw sugar.

In short, we are justified in saying that when making refining crystals or yellow sugars simple defecation and sulphitation are quite sufficient, but that the manufacture of white sugar requires double carbonatation, so that in the present stage of cane sugar manufacture there is no longer any scope for single carbonatation.

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## II.—Lime and Lime Cream.

**Source of the Lime.**

Most factories, which clarify their juice by the defecation process require so little lime that it is not worth while burning it themselves. As the quantity is so small and lime is the only clarifying agent in cane sugar manufacture, it is evident that great care should be taken to use the purest and best available, and it is better to spend a little more money in order to obtain a pure brand than to use a cheap but impure lime, as the difference in expense is so trifling that it cannot enter into comparison with the heavy losses occasioned by the use of bad lime.

As lime rapidly absorbs moisture and carbonic acid from the atmosphere, it is advisable to keep it in closed packages, by preference in soldered tins, but not in baskets, much less in open heaps, as this absorption is very objectionable.

**Quality of a good Lime for Clarification.**

Lime used for clarification should answer the following tests.

When mixed with half its own weight of water it should become very hot within a few minutes; the slaked lime after the addition of ten times its weight of water should form a soft cream, which on being passed through a fine sieve should not leave behind more unslaked particles than one-tenth of the original weight, and most of these particles should become soft after an hour's standing in a moist condition. The lime, after being slaked, should dissolve in hydrochloric acid without appreciable effervescence, and not leave more than 2 per cent. insoluble matter.

The maximum percentages of impurities should be:—

| | Per cent. |
|---|---|
| Iron oxide and alumina | 2 |
| Sulphuric acid | 0·50 |
| Magnesia | 2 |
| Silica | 2 |
| Carbonic acid | 2 |
| Moisture | 2 |

**Impurities of the lime.**

If burnt at too high a temperature, it sinters somewhat and slakes with difficulty or not at all, so that it loses its efficiency. Pure lime, however, requires such a high temperature before it is over-burned, as can hardly be obtained in an ordinary lime kiln, and lime becoming over-burnt at a relatively low temperature is commonly impure. For if the limestone (from which the lime is prepared) contains silica together with alumina or iron oxide, fusible silicates are formed and the lime is very liable to become over-burnt. Usually, the appearance of the pieces of lime indicates, whether it has been over-burnt, and those pieces are generally picked out and removed at the lime kiln, so that the buyer very seldom finds them in the lime supplied him. Water and carbonic acid, though inoffensive, decrease the effect of a given quantity of lime, and the author has, therefore, fixed limits for these and also for the insoluble impurities, which consist of sand or clay. Iron oxide and alumina give rise to the formation of scale in the evaporators, those caused by alumina being remarkable because formed from soluble aluminates during evaporation.

Magnesia is a very troublesome impurity in lime, when present in appreciable quantity. It discolours the juice and retards subsidation, so that the decanted juice always contains much suspended matter, which forms scale in the evaporating vessels. For this reason the maximum allowed should be fixed at 2 per cent. Sulphates also scale the evaporators and should not occur in any appreciable quantities in the lime used for clarification. Alkalies, on the other hand, are harmless and usually only occur in small proportions in limestone, or may be totally absent. A few analyses of lime follow:—

**Analyses of Lime.**

| No. | Sand and Silica. | Iron Oxide and Alumina | Calcium Oxide. | Magnesia. | Carbonic Acid. | Sulphuric Acid. | Moisture. | Undetermined. |
|---|---|---|---|---|---|---|---|---|
| 1 | 0·08 | trace | — | 0·50 | — | — | — | — |
| 2 | 0·10 | 0·44 | 98·08 | 0·63 | — | — | 0·75 | — |
| 3 | — | — | 82·07 | 13·29 | — | — | 4·33 | 0·31 |
| 4 | — | — | 80·19 | 16·38 | — | — | — | — |
| 5 | 0·18 | 0·22 | 95·12 | 0·15 | — | — | 4·29 | — |
| 6 | 0·21 | 0·35 | 96·67 | 0·51 | — | 0·14 | 1·97 | — |
| 7 | — | 0·10 | 97·76 | 0·35 | — | — | — | — |
| 8 | 1·80 | 0·84 | 87·20 | 0·20 | 3·40 | — | — | — |
| 9 | 0·56 | 0·52 | 98·56 | 0·15 | — | — | 0·21 | — |
| 10 | 0·98 | 2·74 | 89·70 | 0·52 | 3·02 | — | 3·04 | — |
| 11 | 0·20 | 0·54 | 87·10 | trace | 4·10 | — | — | — |
| 12 | 0·24 | 1·26 | 89·93 | 0·98 | — | — | 7·59 | — |
| 13 | 0·83 | trace | 91·98 | 5·84 | — | — | 0·90 | 0·35 |
| 14 | 0·12 | trace | 98·65 | 0·69 | — | — | 0·25 | 0·29 |
| 15 | 4·86 | 0·23 | 88·44 | 5·15 | 0·20 | — | 0·71 | 0·41 |
| 16 | 0·14 | trace | 98·48 | 0·72 | — | — | 0·34 | 0·32 |
| 17 | 0·52 | 0·22 | 96·17 | 1·50 | 0·45 | — | 0·90 | 0·24 |
| 18 | 0·12 | 0·20 | 95·68 | 2·08 | 0·52 | — | 0·68 | 0·72 |
| 19 | 0·96 | 1·30 | 91·31 | 1·40 | 1·40 | — | 3·12 | 0·51 |
| 20 | 0·51 | 1·25 | 86·90 | 1·56 | 3·78 | — | 5·44 | 0·46 |

Nos. 3, 4, 13 and 15, are unsuitable for clarification because of their high magnesia content: Nos. 8, 10, 11, and 12, although they do not contain injurious constituents, are yet of inferior quality owing to their low percentage of calcium oxide.

Factories working with the carbonatation process, which require lime as well as the carbonic acid (present in limestone), of course burn their own limestone.

**Selection of the Limestone.**

In the selection of limestone other factors than chemical composition have to be considered now that such large quantities are required, namely, the distance of the quarry from the factory, the means of transport, the hardness of the stone, &c. The harder the stone the more trouble is experienced in breaking it into suitable pieces, and burning it in the kiln. It will therefore be generally more economical to buy a soft limestone found in the vicinity than a hard one which had to be fetched from afar, even if the analysis of the latter is more favourable.

Limestone exists in the forms of marble, limespar, amorphous sedimented stone and coral. The marble and limespar are very pure, but the former often contains a proportion of dolomite (magnesium carbonate) while the latter has the defect of being very hard and very difficult to burn. The coral is a soft stone,

in many cases pure, but sometimes interspersed with terrous strata, which render it unsuitable for burning. Fresh coral from the seashore should be exposed to rain before use, in order to wash out the salt.

**Conditions of a good Limestone.** The maximum impurities found in a good limestone are:—

| | Per cent. |
|---|---|
| Insoluble in hydrochloric acid | 1 |
| Silica | 1 |
| Iron oxide and alumina | 1 |
| Magnesia | 1 |
| Sulphuric acid ($SO_3$) | 0·5 |

The content of calcium carbonate should not be under 95 per cent. Java being the only cane-growing country where the juice is clarified by the carbonatation process, the author can only give analyses of Java limestones burnt in the factory lime kilns, and the following figures refer to limestones from every part of Java.

Nos. 1-9 and 13 are fossil corals, Nos. 10-12 a kind of marble, and 14-15 fresh corals.

**Analysis of Limestone.**

| No. | Sand and Silica. | Insoluble in Hydrochloric Acid. | Calcium Oxide. | Magnesium Oxide. | Carbonic Acid. | Calcium Carbonate | Moisture. | Undetermined. |
|---|---|---|---|---|---|---|---|---|
| 1 | 0·54 | 0·40 | 54·04 | 0·67 | 42·50 | 96·50 | 0·86 | 0·99 |
| 2 | 0·80 | — | 55·35 | — | 43·28 | 98·83 | 0·16 | 0·41 |
| 3 | 0·28 | trace | 54·98 | 0·22 | 43·31 | 98·18 | 0·62 | 0·59 |
| 4 | 0·34 | trace | 55·16 | 0·20 | 43·39 | 98·50 | 0·50 | 0·41 |
| 5 | 0·50 | 0·42 | 54·99 | 0·29 | 43·61 | 98·20 | 0·02 | 0·17 |
| 6 | 6·80 | 2·10 | 50·51 | 0·30 | 40·00 | 90·20 | 0·04 | 0·25 |
| 7 | 0·27 | 0·21 | 54·86 | trace | 42·91 | 97·97 | 0·73 | 1·02 |
| 8 | 0·36 | — | 55·71 | 0·07 | 43·86 | 99·49 | — | — |
| 9 | 0·06 | trace | 55·38 | 0·37 | 43·81 | 98·90 | 0·24 | 0·14 |
| 10 | 0·07 | 0·11 | 42·53 | 11·35 | 45·74 | 75·94 | 0·12 | 0·08 |
| 11 | 0·01 | trace | 55·58 | 0·26 | 43·43 | 99·25 | 0·02 | 0·70 |
| 12 | 0·04 | trace | 55·42 | 0·41 | 44·01 | 98·97 | 0·10 | 0·02 |
| 13 | 0·98 | 0·44 | 54·57 | 0·24 | 42·82 | 97·45 | 0·22 | 0·73 |
| 14 | 0·63 | trace | 54·83 | trace | 43·00 | 97·91 | 1·06 | 0·48 |
| 15 | 0·27 | 0·32 | 54·47 | 0·67 | 43·21 | 97·27 | 0·26 | 0·80 |
| 16 | 0·30 | 0·23 | 54·10 | 0·20 | 42·13 | 96·64 | 1·44 | 1·60 |

On the whole the limestones are pure enough, Nos. 6 and 10 being exceptions: No. 6, a coral, contains too much sand, iron oxide, and alumina or earthy material, and No. 10, a marble, because of a dolomite (magnesium carbonate) vein in the stone. Sulphates could not be detected in any of the samples analysed.

**Moisture Content.** In the best Java limestones the amount of moisture is so trifling that it may be neglected, but in other countries limestones are sometimes so porous and soft, that they may contain very appreciable quantities of moisture. Owing to their firmness the hard limestones may be kept in the open air, but it is advisable to store the more porous ones under cover.

**Fuel for the Lime Kiln.** The fuel used in the lime kiln consists exclusively of coke, of two kinds, viz., foundry coke and gas coke. The following analyses show that the former is the purer, containing less ash and less tarry substance, so that, in order to obtain the same effect, 20 per cent. more gas coke is required than foundry coke, but this difference is counterbalanced by the much lower price of the former.

| | Foundry Coke. Per cent. | Gas Coke. Per cent. |
|---|---|---|
| Ash .. .. .. .. .. .. .. .. .. .. | 8·20 | 19·90 |
| Moisture.. .. .. .. .. .. .. .. .. .... | 0·68 | 2·34 |
| Loss on heating in a current of nitrogen .. | 1·44 | 2·96 |

**Storing Coke under Cover.** When exposed to moist air, coke loses a part of its fuel value, and it is therefore best stored indoors. Care must also be taken not to store the coke in a moist condition, as owing to its porosity it is able to absorb much water, which causes trouble in the kiln owing to the formation of poisonous carbon monoxide.

**Lime Kilns.** The kilns used in sugar factories are of the shaft and Belgian types, fed with mixed limestone and coke. The fact that the burnt lime is thereby contaminated with the ash of the coke has long ago been recognised as of no importance, since this ash is almost totally insoluble both in water and lime-cream, and thus cannot impart impurities to the juice. The burnt lime contains about 2 per cent. ash derived from the coke, the greater part of which forms a fine powder on the surface of the lumps of lime, and falls off during the handling of the lime without causing any trouble during manufacture.

Generator kilns, (in which the lime is heated by the flame from inferior fuel burnt in special furnaces, so that no ash or tar can contaminate the lime), are not used in cane sugar factories, so that our choice is exclusively confined to coke, which does not contain much tarry substance and is hard enough to stand the pressure of the limestone, and not become pulverized, as is the case with many kinds of coal.

**Shaft Kiln.** The shaft kiln is a truncated cone of masonry, lined on the inside with fire brick. The top terminates in an iron funnel closed by a cone, which may be raised or lowered by moving a lever. When charging the kiln, the cone is lowered, and the charge falls through the space between the cone and the funnel into the interior of the kiln, the bottom of the latter being provided with three large openings closed by iron doors, through which the burnt lime may be withdrawn from time to time. The openings for air are

also in the bottom, while the temperature of the different parts of the kiln may be inspected through sight-holes at various heights, which can be closed with plugs. Finally a flue, built of masonry, at a height about three-quarters up the kiln, is connected with the gas suction pipe, conveying the gas to the carbonic acid pump and washer.

**Belgian Kiln.**

The Belgian kiln is made of fire-brick, cased with iron plates, whilst the space between the brick and the outer casing is filled with non-conducting material to retain the heat. It has the form of two obtuse cones, joined together at their bases. The upper and longest cone is provided with an iron funnel and cone at the top (similar to the shaft kiln), which is opened to allow the introduction of fresh charges. The undermost inverted cone is open and its lower edge is about two feet from the ground, the kiln being suspended in an iron ring supported by six iron columns. Under the bottom opening is built a small cone of masonry upon which the burnt lime falls from the kiln. The fallen lime partially obstructs the opening between the kiln and the ground and thus prevents more lime from dropping. From time to time the cooled pieces of lime are removed, allowing the contents of the kiln to fall a little and these, in turn, are removed as soon as they are sufficiently cool.

**Firing the Kiln.**

The kiln should be fired a few days prior to the grinding season, since it takes some time to become sufficiently heated to yield good lime and carbonic acid. To this end a wall of coarse pieces of limestone is made round the bottom of the Belgian kiln and the cone of masonry in order to support the firewood, which is thrown in at the top. In the case of the shaft kiln the doors are merely closed, no wall of limestone being required. Some two tons of burning firewood having been thrown into the kiln, a layer of limestone is spread upon it in order to distribute the flame of the fire evenly throughout the kiln. Above this is added a layer of coke and then alternately layers of coke and limestone in their usual proportion. The funnel is left open until the wood is all consumed, and the lowest strata of the kiln aglow, after which the funnel is closed with the iron cone, and the carbonic acid pump started, the latter drawing off the gases and producing a vacuum in the kiln. This causes air to flow in from below, when it passes through the red hot stones and coke and thus maintains the draught necessary for combustion.

**Charging the Lime Kiln.**

The usual charge consists of 1 part by weight of coke to 9 parts of limestone and, as the weight of the same volume of limestone is thrice that of coke, the proportion of the two materials by volume is $= 1 : 3$, so that for every 3 baskets of limestone 1 basket of coke is added. These materials are commonly hoisted up in baskets containing 100-120 pounds of limestone and thrown down into the kiln through the funnel at the top. It is advisable to alternate regularly the two constituents in order to obtain a homogeneous mixture in the kiln and not local heaps of either limestone or of coke.

This proportion of 3 parts of stone to 1 part of coke is not intended as a fixed one, but may be varied according to circumstances, for example, the size of the pieces of stone or coke, the hardness or softness of the stone, and the quality of the coke. As will be shown later on, the analyses of the gases and the temperature of various parts of the kiln will give sufficient indication as to whether it is necessary to add more of one or other of the ingredients.

**Size of the Limestone and Coke.**

The limestone should be broken into pieces of the size of one's fist and the coke in pieces of half that dimension. Though many mechanical breakers have been invented, it is advisable to break limestone and coke by hand in order to prevent too much waste. If the pieces of limestone are too big they burn slowly, if too small, the draught in the kiln is obstructed and the lime does not slake well afterwards. It is evident that in case of a soft stone pieces may be taken of larger dimensions than when a hard crystalline stone has to be burnt.

When the kiln burns well its contents shrink considerably at the commencement, because of the rapid combustion of the bulky firewood. The charging is continued, always keeping the level of the contents at about one foot below the flue for the carbonic acid. As soon as the contents cease to sink rapidly, the layer of unburnt stones is withdrawn at the bottom. When no more unburnt limestone falls out but well burnt lime appears at the doors or at the bottom of the kiln, the regular working of the kiln commences, all that is necessary is to continue charging the kiln with limestone and coke and to remove the burnt lime, keeping the pump working steadily to maintain draught and combustion. The lime should be removed from below every three or four hours, and the kiln then be charged with alternate layers of stone and coke, always keeping the level of the charge one foot below the suction pipe.

**Theory of the Lime Kiln.**

The carbonic acid is formed both from the combustion of the coke and the decomposition of the limestone, which, under the influence of heat, is converted into quicklime and carbonic acid. Calcium carbonate when heated to 1000° C. becomes totally converted into calcium oxide and carbonic acid, provided this latter is allowed to escape; if, on the contrary, the carbonic acid is not removed, the decomposition stops at a point depending on the pressure of the carbonic acid. In order to obtain a rapid decomposition of the limestone it is therefore necessary to carry off the carbonic acid as fast as it is formed, the more so because cold lime rapidly absorbs carbonic acid to form calcium carbonate once more.

The oxygen of the air combines with the carbon of the coke to form carbonic acid, while the nitrogen remains inactive and passes into the combustion gases unchanged. The water vapour in the air acts upon the red hot coke with the formation of hydrogen and carbon monoxide, which two gases are further burnt to form water and carbonic acid, provided they are not carried off unburnt by too strong a draught, and thus pass into the saturation gas.

By admitting an excess of air so that the saturation gas contains about 1½ per cent. of oxygen, the carbon monoxide is generally oxidized, so that the saturation gas does not usually contain more than 0·50 per cent. of this impurity. That portion of the water vapour which does not attack the carbon acts favourably on the decomposition of the limestone as the temperature at which calcium carbonate decomposes is lowered by the presence of water vapour.

**Carbon Monoxide.**

When the temperature rises considerably above 1300° C., the carbonic acid is decomposed into carbon monoxide and oxygen, both of which are then found in the saturation gas.

The hot gases passing upwards through the thick layers of coke and stone are gradually cooled down to a temperature of about 200° C. The mixture is then washed and cooled before being pumped off to the saturation tanks, because, as we have seen, carbonatation requires a pure and cool gas in order that no impurities be added to the juice and to keep the temperature from rising above 55° C. The composition of a good saturation gas may be represented as follows, calculated on the dried and therefore anhydrous gas.

**Saturation Gas.**

| | Per cent. by volume. |
|---|---|
| Carbonic acid .. .. .. .. .. .. .. .. .. | 28·5 |
| Nitrogen .. .. .. .. .. .. .. .. .. .. .... | 69·5 |
| Oxygen .. .. .. .. .. .. .. .. .. .. .. .. | 1·5 |
| Carbon monoxide .. .. .. .. .. .. .. .... | 0·5 |
| | 100· |

The temperature at which decomposition of the limestone takes place ranges between 1050° and 1300° C. at which complete decomposition may be expected without fusion of the lime. This temperature prevails only in a comparatively small part of the kiln, so that the stone passes through it in about 6 hours, which is sufficient. In order to obtain this, the mixture of limestone and coke must be heated from below by the escaping gases before entering into the zone of decomposition, while after having passed it the burnt lime has still to travel a long distance before it becomes sufficiently cooled by the draught that it may be handled without danger. Experience shows that the depth of this cooling area must be equal to the volume of the limestone burnt in 24 hours, so that the lime requires 24 hours to pass through it. The heating zone and the combustion zone are both half the size of the cooling zone, so that the capacity of the kiln must be equal to twice the volume of the limestone to be burnt in 24 hours.

It is evident that in regular work the combustion zone should occupy the same part of the kiln, *i.e.*, a little more than half way up the kiln. The sight holes in the wall permit the observation of the interior and of the real position of the combustion zone. If this sinks, red-hot lime falls out on discharging, and if it rises, flames escape from the aperture at the top, which, besides placing the

workmen in charge of the filling in imminent danger, may destroy the iron tubes and the funnel, and so render the kiln unfit for use. The right proportions in the kiln can be restored by either sucking off the gases more slowly or more rapidly, and by changing the proportions of fuel and limestone, the operations being always controlled by the analysis of the gas. But, if once a kiln works irregularly, it requires days of strenuous effort to get it into proper order again.

The following irregularities may occur:—

**Irregularities in the Working of Lime Kiln.**

(*a*) Sintering, caused either by impure raw material or too high a temperature or too prolonged a stay in the hot zone caused by a too tardy withdrawal of the already burnt lime.

In the case of limestone containing too much silica, iron oxide, and alumina, or the coke containing too much ash, there is a possibility of more or less easily fusible double-silicates being formed. The ash of the coke chiefly consists of infusible aluminium silicate, but in the simultaneous presence of iron oxide and alumina, double silicates with lime may be formed, especially if the temperature rises above 1300° C. Besides, the lime may also combine with constituents of the fire-bricks forming with them a vitrious fusible combination. This formation of double silicates is the cause of the so-called "scaffolding" of a kiln, while in such a case the lumps of lime become covered with an impermeable layer which prevents proper slaking.

**"Scaffolding" of the Kiln.**

When these phenomena are observed we have either to employ a purer limestone or try to reduce the temperature of the kiln by mixing less fuel with the limestone. If for one or other of these reasons the lime fuses into a solid mass, so that the downward motion is obstructed, no lime falling from the discharge openings (a phenomenon which bears the name of scaffolding), it is necessary to try to break the solid mass by poking it with long irons through the sight-holes and from below.

(*b*) Unburnt lime.

This can arise from too small a proportion of fuel in the charge, insufficient to produce the heat necessary for the total decomposition of the limestone; but it may also be due to the kiln being too small for the amount of work required of it. This can be remedied, accordingly, either by mixing more fuel with the limestone or by building a larger kiln or, if possible, by trying to clarify with a little less lime.

**Unburnt Lime.**

When unburnt lime comes out, together with coke, on discharging, and especially if this is the case at one particular side of the kiln, this indicates that some part of the kiln has cooled down; usually this is occasioned by a cold wind blowing against one side of it and thus cooling the contents locally to a point below the temperature of decomposition. This can, of course, be remedied by erecting a screen or a wall to protect the kiln from the prevailing winds.

(*c*) Red-hot lime.

**Discharge of Red-hot Lime.** In this case the combustion zone has sunk too low and the cooling zone has accordingly become insignificant. By leaving the lime longer in the kiln, the combustion zone rises again, and can further be assisted by accelerating the speed of the pump and thus cooling the lower strata more efficiently.

(*d*) Overburnt lime.

As already stated lime can be overburnt by being exposed to too high a temperature which causes the surface to fuse and to become impermeable to water, **Overburnt Lime.** or it may become coated with an impermeable layer of impurities, which are more or less fusible when in contact with lime. We have already learnt to consider silica (both free and in combination with iron oxide or alumina) as a dangerous constituent in this respect, and when a kiln, which has not been overheated, regularly yields overburnt lime, it is advisable to try whether a purer limestone will not produce better results.

(*e*) Excess of carbon monoxide.

When the gas, passing from the kiln, contains more than one per cent of carbon monoxide, it is a proof that the draught is not sufficient and the speed of **Defective Composition of the Gas.** the pump must be accelerated in order to promote the combustion of the carbon monoxide in the higher strata by a more abundant supply of oxygen. Sometimes, however, even with a sufficient draught, the content of carbon monoxide becomes too high, and this is the case when the coke is too moist, *e.g.*, by being stored after having been in the open and exposed to rainfall and thrown into the kiln without previous drying. The large amount of water vapour in contact with the red-hot coal gives rise to a kind of water gas, consisting of hydrogen and carbon monoxide and thus yielding an impure saturation gas contaminated with the poisonous carbon monoxide. In this case a better draught will not improve matters, and the only remedy is to dry the coke before using it.

(*f*) Excess of free oxygen in the gas.

The gas from a lime kiln in good condition must contain a small amount of free oxygen which need not exceed 1½ per cent. If the oxygen content is higher than this, it may be due to too strong a draught, or to leaks in the kiln, or in the suction pipe. As the pump maintains a vacuum in the kiln, air penetrates through the leaks, which not only dilutes the gas but also causes an undesirable cooling of the contents thus retarding combustion.

(*g*) Sulphuretted hydrogen or sulphurous acid in the gas.

When the coke contains sulphur as sulphuret of iron, this element becomes oxidized in the kiln to sulphurous acid, which at the high temperature corrodes the tubes and the pump, especially when the gas is saturated with water in the washer.

**Capacity and Dimensions of the Kiln.**

The size and capacity of the kiln must be adapted to the weight of cane worked up in the factory to obtain the best results. The total height should not exceed 30 feet, otherwise the weight of the column of limestone and coke becomes too great, and the coke may be crushed. As regards capacity, we usually assume a yield of 16 pounds of lime per 24 hours and per cubic foot of real capacity of the kiln, though of course this quantity may vary considerably according to the hardness of the stone.

One cubic foot of limestone weighs 93 pounds, and requires $\frac{1}{3}$ cubic foot of coke, yielding 51 pounds of burnt lime. We allow the lime to remain for $2 \times 24$ hours in the kiln, so that these 51 pounds of lime require a capacity of $2 \times 1\frac{1}{3} = 2\frac{2}{3}$ cubic feet, and as the kiln is only filled three-quarters full $2\frac{2}{3} \div \frac{3}{4} = 3\frac{5}{9}$ cubic feet total capacity. One cubic foot thus yields 14·8 pounds of lime per 24 hours. When the lime is burnt more quickly, *e.g.*, in $1\frac{1}{2}$ days, the capacity rises considerably, and then every cubic foot yields per 24 hours $\frac{14{\cdot}8 \times 2}{1\frac{1}{2}} = 19{\cdot}6$ pounds of lime, showing that the kind of stone and the varying distribution of the materials considerably influence the capacity of the lime kiln.

93 pounds of limestone yield 51 pounds of lime, and 40 pounds of carbonic acid (assuming a yield of 55 per cent. of lime and 43 per cent. of carbonic acid). Taking the carbon-content of the coke at 80 per cent., and the proportion of limestone to coke as 100 : 11 then $\frac{93 \times 11}{100} = 10{\cdot}23$ pounds of coke, furnish $10{\cdot}23 \times \frac{80}{100} \times \frac{44}{12} = 30$ pounds of carbonic acid, bring the total yield of carbonic acid to $40 + 30 = 70$ pounds. It is evident that for the saturation of the lime obtained, only 40 pounds of carbonic acid are required, but the absorption of the carbonic acid in the saturation gas is very incomplete, amounting to about 60 per cent. of the total, so that only $\frac{70 \times 60}{100} = 42$ pounds of carbonic acid are available where 40 are necessary. This shows that even with such a defective utilization of the carbonic acid, the lime kiln produces an ample supply of this gas, so that we need not fear any shortage. On the contrary it would be possible (in such an emergency, as when the capacity of the lime kiln is too small for the weight of the canes crushed) to purchase some additional lime from other sources, and yet have sufficiently carbonic acid from the kiln to saturate all the lime during carbonatation. When lime is used to the extent of 2 per cent. on the weight of cane, the capacity of the lime kiln should be 300 cubic feet for every 100 tons of cane crushed per 24 hours.

**Methods of Adding the Lime.**

The lime is added to the juice in different ways, generally as lime-cream, but sometimes as powdered quicklime or as slaked lime in the form of a paste.

The pieces of lime are placed in a shallow iron tank and treated with water. It is not advisable to throw the pieces of lime in a tank full of water as they are

**Lime-cream.** thereby cooled too much and do not get properly slaked. After the action is over, the still hot pasty mass is diluted with water, and the lime-cream now obtained is deprived of the sandstones, or unburnt particles, by passing it through several sieves with decreasing meshes. Then the lime-cream is allowed to settle, the supernatant water (which possibly might contain some dissolved matter) is run off, and the remaining lime is diluted to the desired density.

It is necessary to mix the lime with water some time previous to use, in order that the hard particles of lime may soften and become slaked. This is especially necessary in factories using the carbonatation process, wherein large quantities of lime are added to the juice which, although neutralized by the carbonic acid, yet if it contains hard particles these are not attacked by the acid but remain as quicklime in the saturated juice. Afterwards, they gradually dissolve and impart an alkaline reaction to the juice. With single carbonatation, this is not neutralized subsequently, and the concentrated juice is dark-coloured, alkaline, and has a bitter taste due to lime-salts, while this alkaline reaction of the juice with double carbonatation may cause loss of sucrose as an insoluble calcium saccharate.

**Slaking of the Lime.**

The carbonatation process requires a very large plant for the slaking, mixing, and straining of the lime cream, but in the defecation process three slaking and mixing tanks are sufficient.

It is always advisable to use pure water for slaking lime. In factories working with the defecation process the amount of water contained in the lime-cream is insignificant, the quantity so added in 24 hours requiring only seven minutes to evaporate. Factories using the carbonatation process add such a large quantity of water to the juices in the form of 10 per cent. of lime-cream that it is worth the trouble of adopting measures to reduce it. Trials have often been made of utilizing the sweet waters from the filter presses for slaking and diluting the lime, since this water has to be evaporated in any case and might as well be first utilized for preparing the lime-cream and thus save the evaporation of the pure water now used for that purpose. The result is not, however, satisfactory, for if the lime-cream made from lime and sweet water is allowed to stand for a couple of hours the supernatant liquid contains a dirty brown scum consisting of products of decomposition of the reducing sugar in the sweet water by the action of the strong lime. These scums are viscous and render the juice thick, and so retard the saturation. Strangely enough, lime-cream diluted with sweet water can sometimes be used for weeks without any trouble, but suddenly becomes foul (for reasons unknown) and doubles the time required for saturation. This waste of time by unsatisfactory saturation is of course as unfavourable to the work as is the long retention of the diluted juice in the evaporators, so that in this case the whole benefit of thus utilizing the sweet water is lost. As soon as the lime-cream is

**Pure Water necessary for Slaking.**

again made from lime and pure water, the saturation is completed in the usual time, and therefore it is advisable always to employ pure water and not to have recourse to water containing traces of sugar.

**Mixing and Straining the Lime-cream.**

The slaked lime, mixed with water to the consistency of a thick cream, is poured through coarse strainers into two tanks which are filled and emptied alternately. When one of these tanks is filled and diluted to the desired density of 15° or 20° Beaumé, this tank is drawn from until empty. A fresh supply is meanwhile prepared in the second tank so as to be in readiness when the first is empty; this ensures only fresh and well-slaked lime being added to the clarifiers.

The contents of the tanks must be kept in steady motion to prevent subsiding of the lime. In factories where only little lime is used, this may be done by stirring the lime-cream with a paddle, but in carbonatation factories the lime-cream is constantly pumped through a pipe which passes near the mixing tanks and returns to the supply tank. The lime-cream is thus forced from this tank, through the pipes and back again. When lime is to be drawn off and added to the juice, a cock in this pipe is opened, otherwise it continues on its way back to the supply tank and is thereby kept in constant motion and no opportunity given for any lime to deposit.

The lime-content of lime-cream corresponding to the readings of the Beaumé hydrometer are as follows:—

**Density of Lime-cream.**

| Degrees Beaumé. | Weight of 1 litre Lime-cream. | Grms. CaO per litre. | Per cent. CaO. | Degrees Beaumé. | Weight of 1 litre Lime-cream. | Grms. CaO per litre. | Per cent. CaO. |
|---|---|---|---|---|---|---|---|
| | Gr. | | | | Gr. | | |
| 1 | 1007 | 7·5 | 0·75 | 16 | 1125 | 159 | 14·13 |
| 2 | 1014 | 16·5 | 1·64 | 17 | 1134 | 170 | 15·00 |
| 3 | 1022 | 26 | 2·54 | 18 | 1142 | 181 | 15·85 |
| 4 | 1029 | 36 | 3·50 | 19 | 1152 | 193 | 16·75 |
| 5 | 1037 | 46 | 4·43 | 20 | 1162 | 206 | 17·72 |
| 6 | 1045 | 56 | 5·36 | 21 | 1171 | 218 | 18·61 |
| 7 | 1052 | 65 | 6·18 | 22 | 1180 | 229 | 19·40 |
| 8 | 1060 | 75 | 7·08 | 23 | 1190 | 242 | 20·34 |
| 9 | 1067 | 84 | 7·87 | 24 | 1200 | 255 | 21·25 |
| 10 | 1075 | 94 | 8·74 | 25 | 1210 | 268 | 22·15 |
| 11 | 1083 | 104 | 9·60 | 26 | 1220 | 281 | 23·03 |
| 12 | 1091 | 115 | 10·54 | 27 | 1231 | 295 | 23·96 |
| 13 | 1100 | 126 | 11·45 | 28 | 1241 | 309 | 24·90 |
| 14 | 1108 | 137 | 12·35 | 29 | 1252 | 324 | 25·87 |
| 15 | 1116 | 148 | 13·26 | 30 | 1263 | 339 | 26·84 |

The temperature should not fluctuate much from 30° C. The lime-cream generally used is so thick that an accurate estimation of the density is somewhat difficult, so some prefer to dilute a sample with its own volume of water and then determine the density with the Brix hydrometer.

The composition of the undiluted lime-cream corresponding to the degrees Brix in the diluted state is given here.

| Degrees Brix in the Diluted Lime-cream. | Grms. per litre CaO in the undiluted Lime-cream. | Degrees Brix in the Diluted Lime-cream. | Grms. per litre CaO in the undiluted Lime-cream. |
|---|---|---|---|
| 9·— | 93·20 | 14·5 | 150·9 |
| 9·5 | 98·60 | 15·— | 156·— |
| 10·— | 104·— | 15·5 | 161·2 |
| 10·5 | 109·— | 16·— | 166·2 |
| 11·— | 114·— | 16·5 | 171·3 |
| 11·5 | 119·1 | 17·— | 176·3 |
| 12·— | 124·3 | 17·5 | 181·— |
| 12·5 | 129·4 | 18·— | 186·9 |
| 13·— | 134·5 | 18·5 | 192·8 |
| 13·5 | 140·1 | 19·— | 198·5 |
| 14·— | 145·7 | 19·5 | 203·7 |
| | | 20·— | 209·— |

**Disadvantages of Lime-cream.** The use of lime in the form of lime-cream has the following disadvantages:—The strength of the cream is sometimes altered by the subsidation of lime; the cream also adheres to the measuring vessels and thus prevents accurate measurement. Moreover, the preparation and handling of lime-cream is a messy operation, and, finally, the water added for diluting it has to be re-evaporated.

**Unslaked Lime.** Attempts have therefore been made to avoid these difficulties by liming the juice with pieces of unslaked lime, as is done in the beet-sugar industry; pieces of lime being thrown into the hot juice where they rapidly dissolve and exert their clarifying action. This mode of working would only be practicable in carbonatation, as defecation requires so little lime that the introduction of a couple of lumps of lime per measuring tank would be sufficient to supply the desired quantity. Lime will not slake in cold juice but remains on the bottom of the tank when the juice is drawn off to the clarifiers. But even with carbonatation the experiments have failed because the lime slakes very incompletely, the juice being cold and the lumps of lime immediately becoming covered with a sticky layer of impurities precipitated from the juice or resulting from decomposition of the reducing sugar which prevents the further slaking and action of the lime. Attempts were made to remedy this by grinding the lime to a

fine powder, the proper amount of which was weighed into a basket, and immersed in the current of the juice, so that the latter carried off the lime powder without the formation of lumps. But, although a much better distribution was attained, the lime did not slake well in the cold juice, most of it remaining unslaked in the form of hard nodules. When the juice is kept in motion and heated, as in the defecation process, the lime will slake in the juice heaters and clarify the juice there, but when the juice has to be treated with carbonic or sulphuric acid previous to its being heated, the lime has not time to become fully slaked nor to act upon the impurities, while the saturation gases do not reach it but leave the quicklime ready to re-dissolve as soon as the juice is heated, and thus spoil the neutralizing effect of the gases.

The advantage of weighing the amount of lime accurately instead of the unaccurate measuring of the lime-cream is also only an apparent one, because this weighed quantity does not necessarily consist of active lime only. When preparing lime-cream, all the unburnt or overburnt pieces are removed, so that only active quicklime enters the cream. But, on grinding the lime, the inactive pieces remain in the pulverized product and are weighed together with the pulverized active lime. This was found to be the case on analysing three samples of ground lime powder, which did not completely slake after boiling 10 grms. with ½ litre of water, but left hard particles. The same thing was observed in practice, the lime not slaking completely, but leaving 15-20 per cent. of inactive residue. The filter-press cakes from such juice repeatedly contained white patches of unattacked lime, while the defecators and juice heaters had to be cleaned more often because of the lime scale deposited on the tubes.

The quantity of lime applied as pulverized lime may be put down as 15 per cent. in excess of that required when in the form of lime-cream. Instead of 4 litres of lime-cream of 15° Beaumé, containing $4 \times 198 = 592$ grms. of CaO, we therefore need 0·65-0·71 kilogrms. of pulverized lime, though, of course, no greater accuracy is secured than when using the measured amount of lime-cream.

The second advantage of having less water to evaporate is also not as great as it appears. When using 4 litres of lime-cream per 1000 litres of juice, the water so added in 24 hours is removed in the evaporation plant in seven minutes, and this represents a decrease in milling capacity of five tons when 1000 tons of cane are crushed per 24 hours.

As these quantities are very insignificant, there is no advantage in substituting the unslaked lime for the lime-cream.

**Slaked Lime in the form of Paste.**

Lime has sometimes been added to the juice in the form of a paste, portions of which were weighed and placed in the gutter through which the cane juice flows to the measuring tanks. After what has already been stated, the advantages of this system are not great, and we advise manufacturers to adhere to the old practice of using lime-cream.

## III.—Other Clarifying and Decolorizing Agents.

The number of clarifying and decolorizing agents recommended for purifying sugar juices is astonishing, and though new patents are brought out every year, the number of these which survive the first tests remains very limited. In 1887 von Lippman* gave a list of the various chemicals that have been proposed, containing 287 substances, which are grouped as follows :—

**General Review.**

| | |
|---|---|
| Acid of sulphur and allied products | 40 |
| Phosphoric acid and allied products | 25 |
| Different inorganic bodies | 23 |
| Oxygen, halogens | 17 |
| Alkalies, alkaline earths | 48 |
| Metals and their combinations | 66 |
| Organic substances | 56 |
| Electrolytic methods | 12 |
| | 287 |

Since this publication a fresh crop of patents has appeared,† the majority of which have passed into oblivion; and really the chemicals and the methods recommended are sometimes so irrational that we are justified in regarding every new suggestion with scepticism, and can in most cases prophesy its failure.

For this reason the author abstains from enumerating all, but proposes to refer to the more rational schemes in order to show their advantages and defects, or to prove that notwithstanding the promises made by their inventors, they do not really merit the praise bestowed on them.

### 1.—ACIDS OF SULPHUR AND THEIR ALLIES.

#### A.—Sulphurous Acid.

The sulphurous acid, which is applied during the clarification of cane juice is obtained by burning sulphur, the use of the compressed fluid sulphurous acid being too troublesome and expensive in tropical countries owing to the transport of the heavy cylinders in which it is compressed. The raw material for sulphurous acid consists of refined sulphur which is practically pure, and as it never contains more than 0·2 per cent. of foreign matter, chiefly earth, it burns without leaving any appreciable residue.

**Preparation of Sulphurous Acid.**

In sugar factories the sulphur is burnt in iron ovens, which may be cooled by means of a water jacket on the top, and the pipes through which the fumes escape being also provided with a water-jacket in order to condense the volatilized sulphur, and thus prevent the narrow pipes from being choked with sublimed

* *Deutsche Zuckerindustrie*, 1887, 1279.

† Von Lippmann in an article in the *Deutsche Zuckerindustrie*, 1909, page 9, extended the numbers of agents for the various items to 58, 35, 41, 40, 79, 157, 158, and 52 respectively, or altogether to 620.

sulphur. The sulphur is placed on an iron ladle, which is thrust into the oven after the sulphur is ignited. From time to time the door of the oven is opened to add a fresh supply of sulphur. If the sulphur does not burn well, the combustion may be accelerated by placing a red hot iron upon it. In some systems air is forced over the sulphur by an air compressor, while in others an injector in the discharge pipe sucks off the gases and causes a draught of fresh air over the burning sulphur. It is very important that the air passing over the sulphur be dry, since moist air promotes the formation of sulphuric acid from the sulphurous acid and oxygen of the air, which corrodes the oven and the pipes.

The damage done by neglecting to dry the air may be shown by the analyses of crusts and scales in the lead and iron discharge pipes of sulphur ovens in which the air has been allowed to enter undried.

**Scales in the Tubes of a Sulphur Oven.**

| Constituents. | Scales from Tubes. | Scales from Tubes. | Scales from an Oven. |
|---|---|---|---|
| Ferrous sulphate | — | — | 68·30 |
| Ferric sulphate | 35·— | 58·15 | trace |
| Copper sulphate | 25·— | — | — |
| Lead sulphate | 0·90 | — | — |
| Iron sulphuret | — | — | 1·10 |
| Arsenious acid | 1·20 | — | — |
| Free sulphur | 5·80 | 12·72 | 20·77 |
| Free sulphuric acid | — | 12·20 | 1·25 |
| Iron oxide | 4·70 | — | — |
| Water of crystallization | 7·20 | 14·78 | — |
| Hygroscopic moisture | 20·20 | | 6·64 |

The air is dried by passing it through a box provided with trays filled with lime. As soon as the lime is saturated with water on one of the trays it is removed and replaced by another one containing fresh lime, so as always to have an abundant supply of hygroscopic material in the current of the air. When the air is forced over the sulphur by means of an air compressor, it is advisable to have this compressor between the drier and the sulphur oven, for if the drier is between the compressor and the oven the fire will become extinguished whenever the drying box is opened for changing the trays of lime, as the air supply to the oven is then stopped.

**Drying the Air.**

**Composition of the Sulphur Fumes.**

The sulphurous acid content of the gas passing out of the oven cannot exceed 21 per cent. theoretically, but even this figure is never attained, the gas usually containing 14 per cent. of sulphurous acid; sometimes it may rise to 16 per cent. or fall as low as 12 per cent. before the fire goes out.

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### B.—Sulphites.

Instead of sulphurous acid, acid sulphites have been used for clarifying cane juice. In Icery's process sulphurous acid was combined with lime in wooden troughs, and the resulting solution of calcium bisulphite used as a clarifying agent. Later on, sodium bisulphite was occasionally tried and also magnesium bisulphite, but these agents have never come into regular use, being discarded after a few preliminary trials.

**Sulphites.**

### C.—Hydrosulphurous Acid and its Salts.

**Hydrosulphurous Acid.**

Hydrosulphurous acid has a much stronger reducing and bleaching action than sulphurous acid and is used in a great number of industries for these purposes.

The first application of hydrosulphites in the sugar industry was made with Ranson's process. Zinc, and later, tin powder, was added to the juice and a current of sulphurous acid passed through it with the object of reducing that acid to hydrosulphurous acid, which would reduce and bleach the colouring matter more energetically than sulphurous acid alone. As the syrup, after treatment, still contained finely divided metallic powder and also dissolved zinc compounds, and, as the reaction between the metal and the acid was not easily affected, this system was very soon abandoned all the more because pure hydrosulphites could be purchased at a relatively low price.

The dry sodium hydrosulphite is prepared by combining zinc, sodium bisulphite and sulphuric acid in molecular proportions, after which the zinc salt formed is decomposed by sodium carbonate after the following formulae:—

$$Zn + 2\ Na\ HSO_3 + H_2\ SO_4 = Zn\ S_2\ O_4 + Na_2\ So_4 + 2\ H_2\ O$$

$$Zn\ S_2\ O_4 + Na_2\ CO_3 = Na_2\ S_2\ O_4 + Zn\ CO_3.$$

The sodium salt is crystallized out from the solution by adding sodium chloride or bisulphite in the form of hydrated hydrosulphite, having 2 molecules of water. In this state it is very unstable and cannot be deprived of the water by drying without decomposition. It is dehydrated by heating it with absolute alcohol, which combines with the water at 52° C., the mixture undergoing considerable contraction. After being kept at 65°-70° C. for a couple of hours, the salt is filtered off, washed with strong alcohol and dried at 50°-60° C. in vacuo.

**Sodium Hydrosulphite, or "Blankit."**

The salt is a fine white powder, having an acrid odour. When heated on platinum foil it gives off vapours of sulphurous acid and leaves sodium sulphuret. It dissolves easily in water to form an alkaline liquid,* which yields white precipitates with barium and calcium chloride, decolorizes ferric chloride by reduction, reduces potassium bichromate to green chromic sulphate, yields a yellow precipitate with lead acetate, a black precipitate with silver and mercury solutions, and decolorizes solutions of iodine in potassium iodide, and of indigo in sulphuric

* The alkaline reaction is not easily ascertained since the strong bleaching preparation destroys the colour of the test paper and thus renders the reaction indistinct.

acid. The blue colour of the latter reagent is restored after being exposed to the air.

Sodium hydrosulphite oxidizes very rapidly, forming sulphite and sulphate when moist, and still more rapidly in solution, so that it must be stored dry in drums or barrels and not in solution. It is advisable to purchase the hydrosulphite in such small packages that one is sufficient for four or five days only. In this way it only remains exposed for a short time to the moist atmosphere and can be entirely used up before it begins to deteriorate. The proper charge per tank of syrup is weighed out, care being taken to use a dry spatula for this purpose.

The charge stated by the manufacturers is 1 part of hydrosulphite per 10,000 parts of dry sugar obtained, but in many instances double that amount has been necessary to obtain a good decoloration.

Sodium hydrosulphite has been introduced into the sugar industry by the Badische Anilin und Sodafabrik of Ludwigshafen under the name of "Blankit." The French preparation called "Redo," is calcium hydrosulphite, and is claimed to possess the same properties as Blankit, but others report that its action is inferior and that it deteriorates more quickly.

### D.—Action of the Sulphurous Acid Preparations.

Many researches have been conducted regarding the action of sulphurous acid on sugar juices, and most of the theories brought forward have proved contradictory. Sulphurous acid is used for neutralizing, bleaching, disinfecting, and for reducing the viscosity of molasses, and it is well to distinguish those properties when discussing the action of this acid.

#### Neutralizing Action of Sulphurous Acid.

Sulphurous acid being cheap and easily applied is the recognized agent for neutralizing alkalinity in limed juice. Moreover, the lime-salts of this acid are practically insoluble in neutral or alkaline solutions. When it is required to precipitate gums, the juice is heavily limed when cold, and after the lime has precipitated the gums, the excess is neutralized by allowing a current of sulphurous acid to pass through the juice until a drop placed upon a strip of phenolphthalein paper only produces a faint pink tint. Then the supply of sulphurous acid is stopped, and the neutralized juice is pumped through the juice-heaters to the clarifiers.

**Neutralizing Action.**

Phenolphthalein paper should be used for the determining the neutral point, not litmus paper, because even acid sulphites may give an alkaline reaction to red litmus paper, and this would therefore indicate an alkaline reaction when the lime is completely neutralized, and even when part of the lime salt has been redissolved as calcium bisulphite. This would lead to much trouble, and both lime and sulphur would be wasted. Moreover, the dissolved bisulphite decomposes during evaporation, forming normal sulphite and free sulphurous acid. The sulphite is deposited on the tubes of the

**Exact Point of Neutrality.**

evaporators as an incrustation which hinders the transmission of heat and evaporation, while the free acid passes into the condensed water which is used for feeding the boilers, and has a very deleterious effect on the boiler plates owing to its acid reaction. Sulphurous acid, in the presence of moist air, also corrodes the copper tubes at the top of the evaporators, causing them to leak. As all these inconveniences are avoided by leaving the juice faintly alkaline, there is absolutely no reason for continuing the sulphitation after the juice shows a very faint pink tint with phenolphthalein paper.

**Simultaneous Saturation with Carbonic and Sulphurous Acids.**

Several factories, working with the carbonatation process, use sulphurous acid for saturation, and some apply it in conjunction with carbonic acid in the first saturation, while others (the majority) use a very small quantity of sulphurous acid at the end of the second carbonatation in order to neutralize the last trace of alkalinity which cannot be removed by carbonic acid (potash alkalinity).

When using carbonic and sulphurous acids simultaneously in the first saturation, care must be taken never to allow the juice to become neutral but always to keep it alkaline. Calcium carbonate is quite insoluble in neutral or alkaline solutions (using phenolphthalein paper), and calcium sulphite is sufficiently soluble therein to impart an alkaline reaction to litmus paper. In case the solution also contains alkali sulphites, the sugar solution no longer contains any free lime and is therefore neutral from a chemical point of view, but has an alkaline reaction to litmus owing to the dissolved sulphites. If, now, one tries to neutralize this by a further addition of sulphurous acid, the only effect would be to form bisulphites and re-solution of the already precipitated lime salts with all its troublesome consequences. It is, therefore, preferable to use phenolphthalein paper for the determination of the reaction of the juice and to stop the sulphurous acid supply as soon as the precipitate settles well and the juice is still alkaline. In this way the sulphurous acid greatly assists the action of the carbonic acid and cannot do any harm by re-dissolving the already precipitated lime salts.

When using sulphurous acid for the neutralization of filtered juice from which the precipitate of calcium carbonate has been removed, there can of course be no question of re-solution of lime salts and in these cases we can add sulphurous acid more freely and proceed even to a slightly acid reaction to litmus paper.

**Calculation of the Lime necessary for Neutralization.**

In calculating the amount of lime necessary for the common sulphitation of defecated juices we shall assume that the work is done rationally, *i.e.*, that no more sulphur is used than is necessary. Assuming that 1¼ gallons of lime-cream are just sufficient for 500 gallons of juice according to the test on page 130, we take three gallons (or 1¾ gallons in excess), and this 1¾ gallons contain 2·35 pounds of lime which must be neutralized.

$$\underset{56}{CaO} + \underset{64}{SO_2} = CaSO_3$$

56 parts of lime require 64 parts of sulphurous acid produced from 32 parts of sulphur, therefore 2·25 pounds of lime are neutralized by the fumes of $\frac{2{\cdot}25 \times 32}{36}$ 1·3 pounds of sulphur. Say that 100 parts of cane yield 90 parts of juice, then 6000 pounds of cane require 1·3 pounds of sulphur or 0·02 per cent. allowing for a considerable loss of fumes, certainly not more than 0·05 per cent.

Bleaching Action of Sulphurous Acid.

The second property of sulphurous acid is its bleaching or decolorizing power, and here the opinions of various experimenters are divided. Some ascribe the bleaching effect solely to the action of a mineral acid, and claim that hydrochloric and sulphurous acids perform the same service. This is partly true since acid juices always have a lighter colour than neutral or alkaline ones, but in acid solution the bleaching action of sulphurous acid is double that of sulphuric acid, so that besides its action as a mineral acid, sulphurous acid has also a bleaching action due to its reducing action. It must be remarked that this last action only occurs in acid solution, and that the colour returns to its original intensity after exposure to the atmosphere.

**Sulphurous Acid only bleaches in Acid Solutions.**

The requirement that juices and syrups must be acid in order that the action of sulphurous acid may be utilized is inconvenient, because acid juices are always liable to inversion. When the sulphitation is conducted carefully this danger is not present, as impure juices contain so much organic salts that they can stand mixing with a considerable quantity of sulphurous acid even at high temperatures before becoming inverted. Sulphurous acid rapidly inverts pure sucrose solutions, but in the presence of salts of organic acids, sulphurous acid and the sulphuric acid (due to its oxidation) liberate organic acids and become inoffensive salts, the inverting power of the free organic acids being very feeble. Only after careless and repeated sulphitation may the whole of the organic salts become decomposed, and only then may inversion be caused by sulphurous and sulphuric acids.

**The Bleaching Action of Sulphurous Acid is only Temporary.**

A good bleaching of syrups or molasses requires a distinct acid reaction, and in order to impart that, such thick liquids have to be agitated by a stirring apparatus, otherwise the fumes would pass through the thick liquid without mixing with it. Such a stirring apparatus therefore saves both time and sulphur.

Although sulphurous acid is a good bleaching agent for syrup and molasses, it is well not to rely too much upon it but to cure the sugar thoroughly when making white sugar, for the colour returns after some time, so that if the sugar crystals are not freed from adhering molasses they will become tinted and lose their value. It is therefore advisable to do as much as possible to the juice during defecation or carbonatation, and to leave as little as possible to be done subsequently to the syrups or molasses. The acid syrups and molasses possess

the unpleasant property of dissolving metallic substances from the pans and tanks in which they are stored. This is the more objectionable because iron compounds impart a dark colour to sugar, crystallizing from masse cuites containing traces of iron.

**Hydrosulphites also Bleach in Neutral Solutions.**

Hydrosulphites do not require an acid medium but bleach quite as well in neutral or alkaline solutions and are therefore to be preferred to sulphurous acid. The decolorization brought about by hydrosulphites is also unstable, the colour of the bleached juices returning on exposure to the oxygen of the air and for this reason it is advisable to apply this agent at as late a period of manufacture as possible, viz., in the vacuum pan, a few moments before graining.

Decrease in Viscosity effected by Sulphurous Acid.

It is very often asserted that sulphurous acid causes a notable decrease in the viscosity of syrups and thereby a better output from the after-product masse cuites. It is not easy to see how sulphurous acid could have this effect, and careful experiments, conducted both in cane and in beet sugar factories, have failed to disclose such an influence, nor is it known which viscous constituent is precipitated and thus eliminated by sulphurous acid.

**Viscosity.**

Some claim that the sulphurous acid breaks up viscous calcium salts, but it is not clear how such an acid could precipitate lime salts from an already acid syrup, and moreover a direct experiment has shown that sulphurous acid does not throw down or break up lime salts, as will be seen in the following two molasses analysed before and after sulphitation, where the amount of lime salt has not been affected by that operation.

| | Brix. | Ash. | CaO. | CaO in the Ash. |
|---|---|---|---|---|
| After sulphitation .. .. .. .. | 59·14 | 3·54 | 0·504 | 14·2 |
| Before sulphitation .. .. .... | 50·34 | 3·31 | 0·510 | 15·4 |
| After sulphitation .. .. .. .. | 59·34 | 3·40 | 0·426 | 12·5 |
| Before sulphitation .. .. .... | 53·94 | 3·76 | 0·420 | 11·2 |

Some authors even ascribe the property of decreasing the viscosity to sodium hydrosulphite, but this is quite incredible. Moreover, the amount added is very small, and it is at once oxidized to sulphite and sulphate, neither of which possesses any action on the viscous constituents of the juice, so that it is not probable that hydrosulphite can exert such an influence.

Finally, we have to consider the preservative action of sulphurous acid which some authors consider as very strong, since, according to them, juices and syrups are less liable to fermentation after sulphitation. But the author does not attach much value to this property, since with rapid working no fermentation or inversion by micro-organisms is experienced without sulphitation, so that this agent is not indispensable to that

**Preservative Action of Sulphurous Acid.**

end. The sugar, when sold, contains very little sulphurous acid, and as this is very quickly oxidized to sulphuric acid, it cannot prevent deterioration brought about by germs or fungi.

All things considered, sulphurous acid is an excellent agent for neutralizing an alkaline reaction in juices, and it can also neutralize a potash alkalinity after carbonatation. The free acid, and its acid salts, bleach the dark coloured juice in acid solutions, while hydrosulphite can do this also, although the effect is much stronger in alkaline and neutral solutions, but the colour returns on exposure to the atmosphere. The power ascribed to sulphurous acid of throwing down impurities and diminishing viscosity is not yet proved by direct experiments, while its preservative property on juice and sugar is also very doubtful.

## 2.—PHOSPHORIC ACID AND ACID PHOSPHATES.

Phosphoric acid and acid calcium phosphate are known under various names such as Ehrmannite, Newlandite, Claryphos, Clarine, &c. All these are either concentrated solutions of phosphoric acid, or dry or pasty superphosphates of lime.

The free acid is a thick, yellowish liquid, containing 30-40 per cent. of anhydrous phosphoric acid in the form of free ortho-phosphoric acid and a small amount of iron and calcium phosphate.

**Composition of the Phosphoric Acid Preparations.**

The pasty superphosphates contain 25-30 per cent. of free and half combined phosphoric acid and about 10 per cent. of phosphoric acid combined with lime.

A few analyses of these materials follow.

| | | I. | II. | III. |
|---|---|---|---|---|
| | | Soluble Phosphoric Acid. | Ehrmannite. | Ehrmannite. |
| $CaH_4P_2O_8$ | soluble | 2·27 | 28·19 | 38·18 |
| $Fe_2(H_4P_2O_8)3$ | soluble | 7·75 | 12·74 | 2·63 |
| $H_3PO_4$ | soluble | 45·91 | 18·53 | 15·89 |
| $CaSO_4$ | soluble | — | 4·93 | 1·53 |
| $Ca_3P_2O_8$ | insoluble | — | 4·44 | 2·73 |
| $Fe_2P_2O_8$ | insoluble | — | 2·90 | 6·91 |
| Sand | insoluble | — | 0·73 | 1·10 |
| Moisture | insoluble | 44·07 | 27·54 | 31·03 |
| | | 100·— | 100·— | 100·— |
| Free and half combined $P_2O_5$ | | 36·65 | 29·93 | 27·90 |

They are both used for neutralizing an excess of lime, and to this end they are added during the elimination of the defecated or carbonated juice; or they serve during defecation to cause slowly settling precipitates to sink, and are, therefore, added to the juice during clarification together with an equivalent quantity of lime, when the flocculent precipitate of tribasic calcium phosphate carries the impurities to the bottom. In practical working either of these preparations may be used, but it is better to use the free acid for neutralizing purposes and the acid phosphates to produce flocculent precipitates. In the latter case more precipitate is formed for a given amount of free acid, and this is desirable when a heavy precipitate is wanted but superfluous if the aim is only to neutralize an excessive lime content.

**Application of the Phosphoric Acid Preparations.**

The formula given here will show that a given amount of phosphoric acid in the form of acid phosphate will yield 1½ times as much precipitated $Ca_3 P_2 O_8$ as the free acid.

For acid calcium phosphate

$$3\ Ca\ H_4\ P_2\ O_8 + 6\ Ca\ (O\ H)_2 = 3\ Ca\ 3\ P_2\ O_8 + 18\ H_2\ O$$

and from free acid.

$$4\ H_3\ P\ O_4 + 6\ Ca\ (O\ H)_2 = 2\ Ca_3\ P_2\ O_8 + 12\ H_2\ O.$$

Formerly, the phosphoric acid preparations were largely used for neutralizing, but of late they have been replaced by sulphurous acid, which, in fact, is cheaper and more easily applied. This same agent may also serve for promoting subsidation in the settling tanks, and it is therefore probable that phosphoric acid will be eventually replaced by sulphurous acid.

For neutralizing purposes, phosphoric acid is used in a solution of 20° Beaumé, of which 100 c.c. are added per 1000 litres of syrup; while for promoting subsidation, 50 c.c. of that solution and an extra ½ litre of lime-cream are added per 1000 litres of raw juice.

## 3.—ALKALIES AND ALKALINE EARTHS.

(*a*) Soda.

It happens in many cases that cane juice has an acid reaction, although sufficient lime has been added to precipitate all impurities. It is not advisable to evaporate such acid juices, and they should therefore be neutralized, which may be effected during defecation or afterwards on elimination of the settled juice. Until a few years ago, lime was exclusively used for this neutralization, but as lime salts always cause more trouble during the subsequent operations than soda salts, soda has been adopted for this purpose. For purifying purposes lime is, of course, the indispensable agent.

Some use a solution of caustic soda of 20° Beaumé, but others prefer a solution of Solvay soda of the same concentration. A disadvantage of the

carbonate (Solvay soda) is that it effervesces on coming into contact with very acid juice, but this is counterbalanced by the advantages of its being cheaper, more easily handled, not attacking the hands and clothes of the workmen, and not causing any alkaline reaction if by chance it is used in excess, as caustic soda does, but only precipitating a little more lime from the juice, which remains neutral.

(*b*) Barytes.

This agent has frequently been recommended as a universal clarifying substance, and though it is true that it precipitates from the juice some products of the decomposition of glucose, forming with them salts of baryta, these combinations settle very slowly and necessitate a thorough filtration of clarified juice or syrup to eliminate all the poisonous barytes compounds from the juices.

We have already seen how difficult it is to filter the defecated juice and this is the reason why barytes was not introduced for a long time into the cane sugar industry.

It seems, however, that when the application of barytes is followed by a good and sufficient sulphitation, all the barytes is precipitated as sulphites and sulphate, leaving the juice quite free from barytes and, at same time, mechanically carrying down the iron compounds and thus depriving it of all traces of iron, which otherwise would have imparted a dark tint to the sugar crystallized from it.

(*c*) Aluminium, calcium, and magnesium chlorides, alum, &c.

All these salts have been successively used for the purpose of assisting precipitation, but none has proved successful, because too much is needed to produce any effect, and they have all eventually been abandoned.

In laboratory researches, and experiments on a small scale, their effect is generally brilliant, but with the vast quantities of juice worked up daily in factories, the proportions become too difficult and the success less apparent. In Lehmkuhl's process, which consists in adding lime and aluminium sulphate to the juice, the case is even worse owing to the large amount of gypsum introduced into the juice which subsequently causes incrustation on the evaporators.

## 4.—OXIDIZING AND REDUCING AGENTS.

Many chemicals have been recommended for oxidizing or reducing the colouring matters in the juice. In contrast to the types just referred to (which combine with impurities and thus have to be added in considerable quantities), these latter will decolorize in small quantities as the amount of coloration to be removed is quantitatively very small.

It must be stated, however, that the chemicals which destroy colouring matter may possibly also decompose sucrose, and are not, therefore, always so innocuous as their advocates claim them to be.

Among a great number of such preparations the following may be cited here:

(*a*) Ozone.

Ozonized air (2-3 mgrms. of ozone per litre) is forced for 2-4 hours through the syrup of about 20 Beaumé, which has been cooled to 25-30° C.; when cold, the syrup is mixed with sulphurous acid, so that an acidity of 0·02 per cent. is reached. Thereupon barytes is added until the liquid is faintly alkaline; it is then boiled, filtered, and evaporated to masse cuite.

(*b*) Peroxide of hydrogen.

Peroxide of hydrogen in 3 per cent. solution is added to syrup with lime-cream, and simultaneously treated with carbonic acid. At the first saturation (alkalinity = 0 12 per cent.) 5 per cent. of the solution is added, on the second (alkalinity = 0·08 per cent.) 2½, and on the third saturation with sulphurous acid, once more 2½ per cent, or a total dilution of 10 per cent. with the solution of peroxide of hydrogen.

(*c*) Barium peroxide.

Cane juice is mixed with 2·5 per cent. of a milk of barium peroxide of 10-25 Beaumé and allowed to stand an hour or more, until the evolution of gas ceases. The liquid is then sprayed under pressure into a vessel with carbonic acid, whereby barium carbonate and sugar solution are produced, which are separated by filtration.

(*e*) Chlorine with acetylene.

The syrups are treated with chlorine in a specially constructed tank, whereby the colour is bleached. In order to remove the excess of chlorine, acetylene is passed through the syrup, forming an insoluble substance, which floats as an oily layer on the surface and may be skimmed off.

(*f*) Permanganate of lime and of alumina.

The raw juice is treated as usual with lime, then saturated twice with carbonic acid, with this difference, however, that the juice which is turbid after saturation, and still contains a certain quantity of lime is filtered, and heated with a solution of permanganate of lime of 25-30 per cent. This must be added whilst the liquid is cold or lukewarm. The permanganate is allowed to act for 25 to 30 minutes, and saturation is proceeded with as usual; towards the end the mixture is warmed in order to remove the bicarbonates which have been formed. The solution is neutralized with acid, so that the juice contains a slight alkalinity. If sulphurous acid is employed the solution shows a distinct acid reaction, which is just sufficient to prevent the masse cuite from becoming darker.

## 5.—POTASSIUM FERROCYANIDE WITH SULPHUROUS ACID.

Sugar crystallizing from syrup containing iron assumes an unpleasant greyish tint, a very small trace of iron being sufficient to give the sugar a grey appearance, and to lower its value compared with whiter sugars. Ordinary reagents fail to detect or to precipitate these traces of iron in the sugar solution, but when it is reduced by means of sulphurous acid to a ferrous condition, ferrocyanide will precipitate it as white ferrous ferrocyanide. The decolorization of sugar juices by ferrocyanides is based on this principle. Syrup is heated to 60°, mixed with a solution of potassium ferrocyanide and sulphitated. The dark colour of the syrup undergoes considerable bleaching while a flocculent precipitate of white ferrous ferrocyanide is suspended in the liquid. This precipitate is filtered off and then absorbs oxygen from the atmosphere and is thereby transformed into the deep blue ferric ferrocyanide, or Prussian blue. The usual charge is 5 grms. of ferrocyanide per hectolitre of syrup, though this quantity is varied according to circumstances. The first condition is not to employ too much of the reagent and the second to filter the syrup carefully so that no traces of the white precipitate pass over into the clarified syrup. If this occurs, and also if the syrup still contains ferrocyanides, these two remain in the crystallized sugar, first in a colourless state, but afterwards (especially if the sugar is stored in a damp place) the white ferrocyanides are gradually transformed into Prussian blue and the fine white sugar assumes a bluish green tint, which is very detrimental to its value because the buyers might suspect it of being poisonous. Of course, Prussian blue is harmless and as it would require strong chemicals to liberate the hydrocyanic acid contained in it, the sugar itself would be first destroyed, but having to deal with the sugar consumers and their prejudices, one must be very careful in the addition of ferrocyanides and the filtration of the resulting syrup in order to prevent the sugar becoming coloured after being stored, and thereby getting a bad reputation.

## 6.—METALLIC POWDERS.

**Zinc and Aluminium.**

Various metallic powders have been tried with a view to reducing the colouring matter in juices and syrups, using metals which easily dissolve in acids, such as zinc and aluminium, or their alloys with copper and tin.

**Aluminium and Tin.**

Besson recommends an alloy of 85 per cent. of aluminium and 15 per cent. of tin called "alactif," which is added to the syrup in a finely pulverized state in charges of one or two grms. per hectolitre of juice. It at once evolves hydrogen with precipitation of alumina, which action reaches its maximum at an alkalinity of 0·26-0·30 grms. CaO per litre. In neutral juices the action soon stops. According to Besson the advantages are:—

Decoloration of the syrup owing to the reducing action of nascent hydrogen, precipitation of gummy matters by the flocculent alumina and, finally, a much more rapid evaporation in the multiple effet due to the effervescence caused by the escaping hydrogen. Furthermore, this evolution of hydrogen prevents the formation of scale in the tubes of the evaporators, and so promotes transmission of heat and evaporation.

Perhaps these advantages may be realized in beet sugar factories, but in the neutral cane juices the development of hydrogen soon ceased and the greater part of the "alactif" added to the syrup was found unchanged at the bottom. The only advantage was that perhaps the small amount of alumina present caused the scums to settle a little more rapidly than without the addition of the alloy.

Others recommend zinc powder soaked in a 1 per cent. copper sulphate solution and thereby covered with finely divided copper. It is claimed that the close contact between the copper and zinc gives rise to an electric current in the syrup in which the zinc-copper mixture is placed, causing a bleaching and purifying action on the juice without attacking the sugar. It is needless to say that even if these results are obtained, the employment of such poisonous materials is to be strongly condemned.

**Zinc Coated with Copper.**

## 7.—ELECTROLYTIC METHODS.

**Principle.** Electrolytic methods of clarification claim to dissociate the salts occurring in sugar juices by electrolysis and to remove the products of dissociation either by dialysis through porous membranes, or by combining them with the metal of the electrodes, leaving the sugar solution pure.

In the first system, the juice is placed in a tank with porous walls suitable for dialysis. Externally, and on one side, flows a current of water in which the anode is immersed, while on the other side is a current of water in which the kathode is immersed. On allowing a current to pass, the electrolytes are dissociated and dialyse through the porous walls more rapidly than the sucrose.

In the second system, the two electrodes are placed in the juice itself, and when the electric current passes, the salts are split up into free acid and alkali metals. The kathode consists of mercury or some other amalgam which takes up the alkali metals, while the anodes of lead or aluminium combine with the acids to form insoluble bodies which may be separated by filtration.

These systems are excellent in theory, but it is evident that the two porous walls and the thick layer of juice, in the first system, would offer considerable resistance to the current, resulting in loss of energy; moreover, the dialysis of the electrolyzed salts does not proceed very fast, so that some sugar will dialyse at the same time and be lost in the waste waters. In the second

**Difficulties.**

system, it will prove difficult to keep the anode clean, especially when the metal used forms insoluble compounds with the acids; the metallic surface becomes inactive, while the unavoidable development of gas will soon bring about polarization.

Apart from the electrolytic action, a purifying action is also ascribed to an electric current, whereby albuminoids, pectin, and colouring matters, are precipitated. This is true, but this purification may be effected just as well, and at a very trifling outlay, by the usual defecation process, so that it is by no means necessary to have recourse to electricity for that purpose.

**Oxidation of Impurities by the Electric Current.**

Finally it is assumed that the oxygen liberated by the electric current oxidizes the organic impurities, transforming them into oxalates and carbonate. The lime added to the juice precipitates these acids and so deprives the liquid completely of all organic impurities. A simple calculation made by Gurwitsch* shows that even if we suppose the organic impurities to combine with lime in a less oxidized form than oxalate or carbonate and thus escape further reaction, so much energy is employed that for the daily treatment of the juice from 500 tons of beetroot an electrical plant of no less than 3000 H.P. is required.

The precipitation of those organic substances as lead or zinc salts is also much too expensive; and the same author mentions that even if only 25 per cent. of the organic non-sugars be removed in that way, it would require the following quantities of energy and anode material for the electrolysis of the juice.

0·366 kilowatt, or about 0·57 H.P. (per 24 hours) and 1·33 kg. of zinc or 4·27 kg. of lead per ton of beetroots; and for the electrolysis of molasses:—

0·385 kilowatt, or 0·60 H.P. (per 24 hours), and 2·25 kg. of zinc or 7·12 kg. of lead per 100 kg. of molasses.

It is obvious that these figures are much too high to allow of practical application.

At the present day even an advocate of electrolytical clarification such as Gurwitsch says (in his article on the subject in *Zeitschr. Rubenzuckerind.*, 1904, 1013) that up to now all the proposed methods have practically failed. He mentions as the only exception a simultaneous electrolysis and sulphitation to form hydrosulphurous acid, which, as we have seen before, possesses a very high bleaching power. Now that hydrosulphites can be bought at a comparatively low price in a pure state, it is much better to obtain them direct from the makers than to try to make them by an expensive and unreliable electrolytic process. Though none of the proposed methods are of real practical value, we shall briefly review them here.

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* *Zeitschr. Rübenzuckerind.*, 1904, 1013.

(*a*) Schollmeyer and Huber's Process.

Juice, previously freed from mechanical impurities, is warmed to 80°, mixed with lime, so that it contains 2·5 grammes CaO per litre, and then passed through four compartments, in each of which there are five electrodes. Of every five electrodes, three are connected with the positive pole and two with the negative pole or vice versa, when the current is changed. After electrolysis carbonatation follows, and requires 26-30 per cent. less lime than would otherwise be the case. No greater yield is obtained, the advantage consists in economy of lime and more rapid work.

When it is stated that these results were obtained with a plant yielding a current of 850 amperes and 5·5 volts in a factory dealing with 400 tons of beet-roots daily, one might readily suspect that here the electrical current has not done very much, and that this reduced amount of lime could have been attained just as well without electrical treatment.

(*b*) Mangano-electric process of Lavollay and Bourgoin.

This process does not require tanks having porous walls, but the juice is treated in iron tanks provided with zinc electrodes. The electric current has a strength of 0·2-1·0 ampere, and a tension of 6-12 volts for every square metre of electrode surface. After being passed through filter presses, the clarified juice is mixed with 0·9-1 per cent. of finely pulverized calcium manganate, heated to 60-80° C., and exposed to the action of the electric current whilst being constantly kept in motion. At regular intervals, the direction of the current is reversed, so as to diminish the polarization. As soon as the juice has assumed a light greyish colour the current is stopped, freshly precipitated calcium or barium carbonate (1 : 100) is added, the liquid stirred, allowed to subside, and filtered. According to the inventors, the calcium manganate possesses the remarkable property of reacting as soon as an electric current is passed through the liquid, in which it is suspended, but this ceases as soon as the current is stopped. The calcium manganate is believed to serve as a transmitter of the oxygen which electrolysis liberates from the water, combining with same to form permanganate, but yielding up this oxygen to those organic substances which oxydize more readily than sugar, being thereby again reduced to manganate.

The author tried this process in a cane sugar factory, a few barrels of manganate being provided by the inventor. The dynamo which supplied the current for the purification of juice (obtained from 600 tons of cane per 24 hours) was of 10 h.p., so that one's expectations concerning the efficiency of this process were not great. However essential this manganate might be in the opinion of its inventor, the results of this experiment were nil, and, on analysis, this material proved to be merely a mixture of lime and manganese dioxide finely ground together. It need hardly be added that this clarification process has not gone further than the experimental stage.

(*c*) Kohlrepp and Wohl's process.

The alkalis are separated electrolytically and the liberated acids then mixed with basic lead or zinc compounds in suspension in the juice, or with lead saccharate. In this process, insoluble electrodes may be used, which, being neither attacked nor affected by deposits, do not require renewing.

The juice is treated with about 0·3 per cent. of calcium oxide and carbonic acid and filtered through presses after the addition of calcium carbonate or other material for promoting filtration. 8-10 per cent. of moist lead saccharate is added, after which air is blown through the mixture which is electrolyzed at a temperature of 60° with constant stirring. The positive electrodes consist of coal, while iron plates form the negative electrodes, and parchment paper serves as a diaphragm.

The lead oxide is afterwards regenerated by decomposing the moist plumbic precipitates with caustic soda. The resulting liquid is evaporated, charred, and the alkali re-extracted for subsequent use, while the plumbic oxide is again combined with sucrose to form saccharate.

The trace of lead compounds remaining in the juice after electrolysis is removed by carbonic acid.

This system is so intricate that it has found no practical application.

(*d*) Say-Gramme process.

Finally, we have the Say-Gramme process, which is kept strictly secret and is reported to work with much success in the Egyptian factory of El Hawamdieh. Here, the juice is slightly limed and passes through three dialysing vessels in which are immersed anodes of magnesium or lead, zinc or iron, so that the liberated acid may find a metal with which it forms insoluble compounds.

Probably the *modus operandi* is other than stated here, but notwithstanding favourable reports received periodically, its application does not extend, and in this respect it shares the fate of other electrolytic processes in cane sugar manufacture.

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## IV.—Filtration.

In cane sugar factories we meet with two different kinds of filters, viz., juice filters for the filtration of juice, and scum filters in which the juice is separated from the juice.

Bone black filters, which only serve to decolorize dark liquors, and do not take out floating substances, are not of use in Eastern countries as they are apt to prevent the use of the sugar decolorized with material of animal origin in countries inhabited by a Hindoo population.

## 1.—Juice Filters.

The simplest form of juice filter consists of a bag made of cloth, into which the juice is poured, the clear juice passing through and the impurities being kept back. Such are the "Puvrez" filters, consisting of a cylindrical bag, of which one end is closed and the other tied round a pipe supplying the juice to be filtered, which enters into the bag under a certain pressure. The impurities are kept back, and the filtered juice passes through the cloth and flows into an iron gutter to which the bag is fixed, and so is conveyed to the suction tank of the evaporators. A great inconvenience of this filtration from inside to outside is that the finer impurities also are driven between the pores of the cloth and choke them up, so that the bag does not filter for long before becoming full of scum, and then has to be changed. Consequently filters of this description have now been generally abandoned, and other systems have been adopted in which the filtration takes place from outside to inside, of which we can mention the Danêck, Kasalowsky, Philippe filters, &c.

In these systems the filter consists of a square iron vessel on which an iron plate is screwed as a cover. In this vessel are a certain number of chambers lined with cloths, kept apart by perforated plates or metal spirals fitted in the chambers. Each chamber is therefore shut off by cloth at the sides and at the bottom, and the upper edges are pressed tightly together, thus preventing the juice, with which the filter is filled, from entering the chamber without passing through the cloth. Each chamber has a spout for the discharge of filtered juice, and a large cock at the bottom of the filter enables the workman to empty the whole of the contents when it has to be cleaned. When starting, the chambers are supplied with new or clean cloths, the filter is closed with the cover, the screws are tightened, and the hot juice is admitted under a head of three to six feet, avoiding great variations. The juice passes through the cloth, leaving behind the suspended particles, which sink to the bottom of the filter and do not interfere with the filtration through the cloth. The latter can therefore be used for a long time, but when it is dirty it is changed for a new cloth, and as soon as the contents of the entire filter have become too muddy owing to the impurities deposited, it is emptied by means of the cock at the bottom; the juice is conveyed to the scum tank, while the filter is washed and made ready for use again.

In many cases, the floating impurities are so fine or gummy that filter cloth is unable to retain them, though much may be done in this direction by keeping the juice hot and maintaining a good pressure in the filters. On this account juice filters have been discarded in most factories, and manufacturers prefer to evaporate their subsided juices unfiltered.

However, there is no doubt that juice, even if allowed to subside for a long time, always contains many floating and suspended particles which deposit on the tubes

**Difficulties during Filtration.**

of the multiple evaporating apparatus. It is those colloids and suspended impurities which choke up the pores of the filter-cloth and hinder filtration, and all attempts to remedy this by the addition of infusorial earth or clay have up to now failed. The filter works well during the first few minutes, but the cloth is soon covered with a slimy layer which clogs the pores of the cloth so that it has to be changed and cleaned.

Among the many substitutes for cloth, bagasse was very popular for some few years, after that bagasse filters were introduced, but these have now been discarded. Bagasse from the second mill was preferred, that from the first mill being too coarse and that from the third being too fine and powdery.

**Bagasse Filters.**

The unfiltered juice enters the filter from below under a head of some 4 or 5 feet, and forces its way upward through the bagasse, leaving its impurities behind and passing out at the top of the filter through a spout or pipe. The bagasse is therefore constantly covered by hot juice which prevents its getting sour. When the bagasse is saturated with scum or dirt the supply is stopped and a cock at the bottom of the filter opened. The unfiltered juice remaining in the filter then flows into the scum tank and the bagasse is transported to the mill, which extracts the juice which it had imbibed and thus avoids the loss incurred in other systems. But the hot bagasse is a difficult material to handle, besides which the carrying of clean and dirty bagasse to and from the mill is an untidy operation.

The only disadvantage from a chemical point of view (viz., the dissolving action of limed juice on the fibre of the bagasse) has not proved very serious, since from the experience gained with Naudet's diffusion process where limed juice passes through cane fibre at a high temperature, it was proved that the masse cuites were not more gummy than by mill-extraction. But even if this were so, it could be avoided by using the smooth fibre of the sugar palm instead of bagasse, although, in this case, the advantage of the cheapness of the filtering material and the recovery of the juice contained in it are lost.

Such a smooth fibre, derived from a kind of aloe, and known as "crin végétal," or vegetable horse hair, is used in the decanting filter of Bouvier.

**Bouvier's Decanting Filter.**

This consists of a rectangular iron tank, 3 feet high, 3 feet broad, and 5 feet long, which is divided into 8 or 10 compartments by iron baffle plates which extend to within a couple of inches from the top and bottom alternately, causing the juice to take a zig-zag course before it reaches the outlet. Those compartments wherein the juice flows upwards contain baskets filled with the vegetable horse-hair filtering material, while the other compartments are empty and have discharge cocks at the lower part of the inclined bottom. The juice enters an empty compartment of the filter, and during its downward course deposits heavy impurities at the bottom. It then enters

the second compartment containing the filtering medium, which holds back the finer impurities. This goes on four or five times, until it leaves the tank completely filtered. The subsided dirt is removed from time to time by opening the cocks at the bottom, the filtering medium when saturated with dirt being removed and replaced by fresh. It is said that four such filters are sufficient for the filtration of the juice from 600 tons of cane per twenty-four hours.

**Sand Filters.** Instead of bagasse or the above aloe fibre, inventors have suggested pulverized cork, but the dirt was not retained by this material. Better results are reported of the sand filters of the Danêck and Abraham types. In both these systems, the juice is forced through a layer of quartz sand, having a large filtering area, and which can, therefore, do more work than cloths. When the filter becomes clogged by a layer of dirt, a new filter-bed may be obtained by simply agitating the uppermost layer of sand. Only when the whole of the sand is saturated with dirt is the juice let off and the sand washed by a strong jet of water. The first washings are transferred to the scum tank, while the later and diluted ones are thrown away.

The sand retains a considerable quantity of slimy material and very fine bagasse fibre in a semi-colloidal state, and, therefore, diminishes the formation of scale in the first vessels of the multiple evaporation plant.

**Composition of the Dirt retained by Sand Filters.** The composition of a sample of slime scraped from a dirty sand filter is as follows, calculated on the dry substance :—

| | Per cent. |
|---|---|
| Cane fibre | 69·1 |
| Albuminoids | 6·9 |
| Wax | 7·7 |
| Ash | 16·3 |
| | 100· |

The ash consists of :—

| | Per cent. |
|---|---|
| Calcium silicate | 5 |
| Calcium phosphate | 8·8 |
| Calcium carbonate | 2·2 |
| Undetermined | 0·3 |
| | 16·3 |

A defect of sand filters is that they require a special kind of sand which is rather expensive, and although the material may be used again, after washing, there is always a certain amount of waste. It is not advisable to use other kinds of sand, which may possibly differ in efficiency and thus spoil the work.

The subsided juice may also be filtered through fine bronze wire-gauze, such as is used for "bolting" flour. This gauze contains about 2000 meshes per square centimetre, the diameter of the wire being 0·093 mm., the width of the meshes 0·137 mm., and the composition 93 per cent. copper and 7 per cent. tin. It does not require any intricate system of filtering chambers, but can be used as a strainer through which the decanted subsided juice is caused to pass. The juice passes very rapidly through this gauze and is received in a tank underneath, leaving a thick layer of fine bagasse fibre and slime on the strainer, which is scraped off from time to time. As the gauze is very delicate and liable to break during cleaning, it is advisable to place the strainer in an inclined position with a strip of copper plate at the lower end, and also to protect the fine gauze by a piece of "birds' cage" gauze, so that the scraper cannot touch the former. The dirt gradually sinks towards the strip of copper plate below, and may be removed from this without damaging the soft fine gauze.

**Bronze Gauze.**

A portion of dirt retained by the wire gauze gave this analysis after being washed and dried.

| | | |
|---|---|---|
| Gummy and fibrous substance | | 95·60 |
| Ash | | 4·40 |
| The ash contained | Silica | 2·54 |
| | Calcium phosphate | 1·30 |
| | Calcium carbonate | 0·57 |

Cane fibre generally contains 2·50 per cent. of ash, of which 1·75 is silica, 0·20 calcium phosphate, and 0·10 per cent. calcium carbonate, so that the dirt retained by the wire gauze also contains an appreciable quantity of inorganic substances, which would otherwise have caused scale in the evaporating vessels. As this strainer is easily fitted up and inexpensive, it is advisable to use one for straining the whole of the juice before it enters the evaporating vessels.

Though not exactly a filter, Hignett's juice centrifugal may be mentioned here. This is intended to separate the defecated, but unsubsided juice by centrifugal force in a continuous manner, yielding a clear juice and a heavy deposit of scum. The centrifugal is made of steel plate, and is not perforated. The upper edge of the casing carries a circular gutter supported on iron brackets, and is connected with a discharge pipe. The basket of the centrifugal has an opening at a lower level connected with another discharge pipe. Defecated juice enters the centrifugal in an uninterrupted stream, the heavy dirt (being lifted higher by the centrifugal action) flows off through the circular gutter, while the clean juice escapes through the lower opening, and is conveyed to the evaporating plant. Experiments with this centrifugal in Java have not been successful.

**Hignett's Juice Centrifugal.**

## 2.—Scum Filters.

The most primitive method of treating scums was by pressing them in closed linen bags which were placed in a box provided with a perforated bottom, the pressure being assisted by a cover loaded with stones. A part of the clear juice is thus forced through the cloth of the bags, after which the remainder is expressed in a press. This operation demands much labour, is dirty, and also unsightly.

**Bag Filters.**

The Taylor filters are a great improvement, and consist of a number of long narrow bags, closed at the lower end, suspended in an iron case furnished with a receiver for the scum. Into the bottom of this receiver are screwed several rows of nipples to which the above-mentioned bags are fixed. The receiver being filled with the scum, the latter runs into the bags, which retain the impurities, while the clear juice is filtered off and flows into the lower part of the apparatus, from which it is conducted by a gutter to the suction tank of the evaporators.

**Taylor Filters.**

The front of the apparatus is provided with a door so as to allow the bags to be changed, but as this work proved troublesome in the hot narrow cases, the upper receiver has been made movable in later patterns, so that it may be hoisted with all the attached bags by means of a travelling conveyor and carried outside the factory. The hot bags can thus be changed out of doors, and while this is being done a second receiver fitted with clean bags is placed in the box so that the filtering can go on without much interruption.

When the bags are full of mud they are detached from the nipples tied round the top, pressed, emptied and washed. Sometimes their contents are washed out by pouring water into the bags but this operation is generally omitted because the water penetrates the slimy dirt very slowly.

As the filtration proceeds from the inside outwards, the same inconvenience is met with as in the "Puvrez" filters, though not to so great an extent, as the pressure is much less and the scum more compact. The disadvantage that the juice cools very rapidly owing to the great surface of bags exposed to the air, and hence does not filter so quickly, or even becomes acid, can be remedied by blowing a constant jet of exhaust steam into the chamber, which keeps up the temperature, prevents sourness, and increases the capacity by 20 per cent.

In place of these filters most factories now use *filter presses*, which consist of metal chambers lined with cloth, and screwed tightly one against the other. A pump or monte-jus, or air compressor, fills them with the hot scum. The juice passes through the cloth and escapes through suitable channels, while the solid matter remains behind in the form of hard cakes. As every chamber has its own discharge pipe, which can be closed by a cock, any of them can be stopped working if the filtered juice should become turbid owing to a hole or leak in the filter cloth.

The oldest filter presses consisted of plates having a central opening through which the juice entered the press and which at the same time served for fastening the cloths. This system was abandoned partly because the continuous fastening and loosening of the screws occupied too much time, but chiefly because the cloths wore away very rapidly at that spot and had to be continually mended. Therefore the inlets for juice, for the wash-water, and for steam were all placed on one side of the plates, but then the holes in the cloths were all on the same side, and leakages could not be prevented. Kroog constructed filter presses in which the inlets for juice, water, and steam, were situated in the upper corners of the plates and were thus outside the actual filtering surface. These inlets were joined by indiarubber rings or by pieces of cloth provided with a circular opening. Beeg's filters are distinguished by the special construction of the frames (with fine corrugations), rendering superfluous the sieve plates which are to be found in other systems. The cloths are fastened by bronze springs, which grip as soon as the press is closed, the grip being greater the tighter the press is screwed.

The scum should enter the filter-press as hot as possible, having an alkaline reaction, and at a pressure of from 1½ to 2 atmospheres. When the chambers are full of scum-cake, as indicated by the fact of no more juice flowing off, the supply of scum should be stopped, the cakes washed with water or steam, the press opened, and the cakes finally removed. The cloths are changed if they have become too dirty, after which the press is again closed and is ready for further use.

**Steaming of the Scums.** The steaming of the scums has the effect of driving the residual juice from between the particles of the cake and does not dilute this juice. Such displacement of juice is only attained when the cakes are thin ; in those one inch thick the effect is very small.

**Washing the Scums.** The washing of the filter-press cakes with water is only applicable in factories using the carbonatation process, because the cakes obtained by defecation are much too gummy to allow of their being washed in the press. It is best to leave them with their large sucrose content or they might be washed in a Ledocte malaxeur, like carbonatation cakes. This is a revolving drum in which the cakes are stirred with water, after which the diluted juice is again filtered in presses. In carbonatation factories, this juice is used as maceration water at the mills, but that from defecation cakes is much too impure for this purpose as it contains a great deal of dissolved impurities. It is advisable to return it to the scum tank, where it is defecated again and then serves to dilute the juice somewhat and make it easier to filter. This, however, may only be done when the evaporating plant can deal with all this water, otherwise it is preferable to leave the cakes unwashed and still containing juice.

It often occurs that defecation scums, even when filtered hot, under sufficient pressure, and with a distinctly alkaline reaction, do not yield hard cakes, filling

**Impossibility of getting Hard Cakes.**

the whole chamber; but soon after the filters have started, filtration stops and the chambers are found to be filled with a thick mud, containing much juice, which goes to waste. This may be caused by wax deposited from the hot scums upon the cold cloths, which become clogged with that substance. In order to avoid this, it is advisable to steam the press before introducing the scums.

The scums may also be too slimy and this is best remedied by using a coarser strainer for the juice, which allows more of the fine bagasse to pass into the juice and renders the scums more porous and capable of being pressed dry. It is, however, advisable not to go too far in this direction since every addition of fibre increases the amount of scums and the loss of juice in them, while the capacity of the presses decreases accordingly.

**Loss of Sucrose in Filter-press Cake.**

The usual sucrose content of unwashed defecation press cake amounts to 9 per cent.; that of washed cake to not more than 4 per cent.; while the sucrose content of washed carbonatation press cakes may fall as low as 1½ per cent. As on 100 parts of cane far more scums are obtained with the carbonatation process, the loss of sucrose on 100 cane in filter-press cake is much the same in both cases, and amounts to 0·06-0·09 per cent. or slightly more or less.

**Utilization of Filter-press Cake.**

The filter-press cake is usually employed as fertilizer.

During the last few years attempts have been made in some Java factories to extract the wax from dried defecation press cake on a commercial scale. The cakes are dried in the sun or on the furnace-flues, extracted with benzine in a special apparatus and the crude green wax bleached with chlorine. But so far no particulars have come to hand as to the success of these experiments.

## CHAPTER III.

# CONCENTRATION OF THE JUICE.

Up to the stage now reached, all the operations the juice has undergone have had for their object the removal of the non-saccharine matters and the clarification of the juice. This being accomplished, the sucrose now exists in a solution containing about 15 per cent. of dry substance, including those impurities which clarification failed to remove. The purpose of the next operation is, therefore, to convert the sucrose into a solid in order that it may then be separated from the impurities still remaining in solution in the mother-liquor or molasses. This is effected by evaporating water from the juice until the small quantity of water left can no longer hold all the sucrose in solution; this latter accordingly crystallizes out and can be separated from the mother-liquor and thus obtained in a pure state.

**Principle.**

This withdrawal of water by evaporation must be made at once and without delay, as in a dilute solution the sucrose is very liable to decomposition by the action of micro-organisms, which cause inversion or sourness and therefore give rise to considerable loss. It is therefore necessary to deprive the clarified juice of the great bulk of its water as rapidly as possible, so as to bring it into a condition in which it is no longer liable to such decomposition, *i.e.*, a crystalline mass containing 8-10 per cent. of moisture.

Since sucrose is liable to decomposition at high temperatures and also inversion, it is desirable to keep the temperature as low as possible during evaporation and at the same time to reduce the time during which it remains at that temperature. This is effected by boiling the juice under reduced pressure, by means of which its boiling point falls, hence the present-day practice of boiling the juice *in vacuo*. Powerful pumps are used to draw off the vapours and maintain a vacuum.

**Concentration in Vacuo.**

The concentration of the juice is ordinarily effected in two stages, viz., *evaporation*, which concentrates the juice to syrup containing about 50 per cent. of solid matter, and *boiling*, which concentrates this syrup still further, and transforms it into a crystalline mass, containing only from 8 to 10 per cent. of water.

**Concentration is performed in Two Stages.**

The sole object of evaporation is to concentrate the juice to syrup as soon as possible, with a minimum waste of steam and sucrose and at a low temperature.

In the boiling process (during which the sucrose first assumes a crystalline form), additional care is necessary in order to obtain the maximum yield of crystallized sucrose in as pure a state as possible, and in such a condition as to allow it to be separated from the mother-liquor with the minimum loss and trouble.

Between these two stages, the syrup is in most cases subjected to a second clarification, which is of considerable importance in the manufacture of white sugar, as it is impossible to obtain good white sugar from a turbid syrup.

**Decomposition of Sucrose during Evaporation.**

Though the evaporating and boiling processes are carried out very rapidly, it is not possible to avoid partial decomposition and caramelization; the juice is liable to become overheated locally, which is seen when a syrup or a masse cuite is diluted to the density of the original clarified juice, and the colours compared, the colour of the former always being darker than that of the latter.

According to Gröger's* investigations, the increase in colour is strongest in the first vessel of the evaporators, *i.e.*, that in which the temperature is highest, and it is much less in the subsequent vessels, in which the juice boils at lower temperatures. This point will be considered in greater detail further on.

**Calculation of the Weight of Water to be evaporated.**

The amount of water to be evaporated may be calculated from the degrees Brix of clarified and concentrated juice by the formula:—

$$W = k_1 \left(1 - \frac{S_1}{S_2}\right)$$

in which

$W$ = weight of water to be evaporated.
$k_1$ = weight of clarified juice.
$S^1$ = Brix of clarified juice.
$S_2$ = Brix of concentrated juice.

---

## I.—Evaporation.

Formerly, the clarified juice was evaporated in shallow pans, known as the "Copperwall," over an open fire, but this process had the inconvenience of utilizing the fuel very imperfectly, and it was also impossible to prevent local overheating, which caused a portion of the sugar to be become decomposed. As the pans were open, and the impurities were constantly skimmed off, the mechanical purification was very good, but this advantage could not counterbalance the enormous losses of sugar and fuel.

**Evaporation over open Fire.**

**Evaporation by Steam under Atmospheric Pressure.**

Later the juice was evaporated in "Concretors," *i.e.*, open pans, heated by steam, which secured a more economical utilization of the fuel, and avoided the risk of overheating, but they too were soon supplanted by the multiple-effect apparatus. In these the juice boils *in vacuo*, and therefore at a lower temperature, so that inversion is no longer to be feared; moreover, the employment of two, three, or even four vessels allows the heat of the steam to be utilized as completely as possible; the vapour from the first

**Evaporation in Vacuo.**

* *Oesterr-Ung. Zeitschr. für Zuckerind. und Landb.*, 1898, 790.

vessel passes into the steam drum of the second, that from the second heats the juice in the third, and so on, as will be seen from the following description.

The evaporating vessels, generally in use, consist of large iron cylinders, with dome-shaped heads, fitted with a wide pipe which carries off the vapours. The lower part of the cylinder is divided into three portions by two transverse plates pierced with holes, in which are fitted tubes which bring into communication the upper and the lower portions of the cylinder, together called the juice-chamber. The middle portion is called the steam drum and is traversed by the tubes, but does not communicate with them. The steam drum of the first vessel is provided with an inlet pipe for exhaust steam, and in most cases with a smaller one for direct steam, also with an escape pipe for the condensed water. The wide pipe which carries off the vapours from the juice is so constructed as to prevent particles of juice from passing off with the vapours, and communicates with the steam-drum of a second similar vessel, and so on, while an air-pump, attached to the vapour pipe of the last vessel, draws off and condenses the steam. The steam-drums are kept free from condensed water by means of pumps.

**Working the Triple-effet.** Each vessel of a triple-effet is filled with juice to the level of the upper tube-plate, so that the entire heating-surface is covered by the juice to be evaporated.

Steam, having a temperature of over 100°, on being admitted to the steam-drum of the first vessel, causes the juice to boil, and becomes itself condensed. The vapour from the juice enters the steam-drum of the second vessel, and heats the juice contained in the juice chamber, where it is under a certain vacuum, and therefore boils at a lower temperature than that in the first vessel. The vapour formed here is similarly capable of boiling the juice in the third vessel, because this, being in immediate communication with the air-pump and the condenser, boils at a still lower temperature than the juice in the second vessel. The vacuum is therefore greatest in the last vessel, and least in the first, in which on the contrary a certain pressure is maintained; consequently the juice in the last vessel boils at the lowest temperature.

The removal of the condensed water from the steam-drum of the first vessel was formerly effected by a syphon about 15 feet in height, as measured from the lowest tube plate, but now that the first vessel is generally worked under pressure, the condensed water is allowed to flow out through a steam trap. The condensed water of the second and third vessels is removed by a pump if it is to be used for feeding the boilers; otherwise, the steam drums are simply connected with the air-pump. They are therefore always kept free from water, and as the vapours from the juice in the preceding vessels are immediately condensed by the cold juice, a vacuum is produced in the juice chambers in the first and second vessels.

In order to remove air and gases from the steam drums, narrow tubes are fitted to each drum, such tubes communicating with the dome of the same evaporating vessel. Though no ammonia escapes from cane juices, these tubes are called "ammonia" tubes from analogy with the corresponding tubes used in beet juice evaporators.

The clarified juice is pumped into a tank, from which it gravitates into the first vessel, or is pumped into it if the first vessel is worked under pressure. It enters at the bottom of the juice chamber, where various arrangements are made to facilitate the mixing of the fresh juice with the juice already present in order to ensure a good circulation. The vessels are connected with one another by means of pipes provided with cocks, by the cautious opening of which the juice is drawn from the first vessel into the second, and from there into the third, by the difference in vacuum of the three vessels; in this way the clarified juice enters the system, and leaves it with the concentration of "syrup." A proper regulation of the cocks permits the supply of juice to equal the evaporated water so that the concentration goes on steadily without changing the position of the cocks.

Care must be taken to keep only such a quantity of juice in the first vessel for the upper tube-plate to be just covered; if the hot plate is not constantly moistened by the juice, overheating is to be feared, and if the vessels are too full, there is a risk of losing juice by its passing off with the current of vapour leaving the vessel. These remarks are also applicable to the other vessels, though with them the danger of overheating is not so great. If they are insufficiently filled, they should be supplied from the other vessels; in the contrary case, the supply is stopped, but the syrup is only drawn off when it has the required specific gravity.

A high temperature being always detrimental, it is advisable to work with as little juice as possible in the vessels and to keep the juice level low in each, though, of course, decomposition cannot be entirely avoided.

**Decomposition of Sucrose during Evaporation.**

Claassen* calculated from Herzfeld's tables the quantity of sucrose decomposed during evaporation. As these are calculated for slightly alkaline juices and cane juices gradually become acid during evaporation, the figures given are not perhaps quite applicable here; but where they are not stated for cane juice by direct experiments, Claassen's actual figures are given.

**Claassen's Experiments.**

Claassen's experiments were made with a low level of juice in the evaporators and concerned:—

1. Quadruple-effet with high-pressure steam.
2. Quadruple-effet with low-pressure exhaust steam.
3. Triple-effet with juice heater using steam under pressure of 1 atmosphere.

* *Archief voor de Java Suikerind.*, 1894, 185.

I. Quadruple Effet with High-pressure Steam.

| No. | Period of Evaporation in minutes. | Temperature in °C. | Sucrose in the Juice. | Loss of Sucrose per hour according to Herzfeld. | Loss of Sucrose on 100 Sucrose during Evaporation of the Juice. | Loss of Sucrose on 100 Beet. |
|---|---|---|---|---|---|---|
| First vessel .. .. | 7 | 112 | 10 | 0·1677 | 0·0195 | — |
| Second ,, .... | 8 | 105 | 15 | 0·1162 | 0·0155 | — |
| Third ,, .. .. | 12 | 95 | 25 | 0·0512 | 0·0102 | — |
| Fourth ,, .... | 27 | 68 | 50 | 0·0100 | 0·0045 | — |
| | 54 | | | | 0·0497 | 0·0065 |

II. Quadruple Effet with Low-pressure Exhaust Steam.

| No. | Period of Evaporation in minutes. | Temperature of the Juice in °C. | Sucrose in the Juice. | Loss of Sucrose per hour according to Herzfeld. | Loss on 100 Sucrose during Evaporation in the Vessels. | Loss on 100 Beet. |
|---|---|---|---|---|---|---|
| First vessel .. .. | 8 | 102 | 10 | 0·1230 | 0·0164 | — |
| Second ,, ... | 11 | 96 | 15 | 0·0844 | 0·0155 | — |
| Third ,, .. .. | 17 | 86 | 25 | 0·0342 | 0·0097 | — |
| Fourth ,, .... | 43 | 65 | 50 | 0·0100 | 0·0071 | — |
| | 79 | | | | 0·0487 | 0·0063 |

III. Triple Effet with Low-pressure Steam and Juice Heater.

| No. | Period of Evaporation in minutes. | Temperature of the Juice in °C. | Sucrose in the Juice. | Loss of Sucrose per hour according to Herzfeld. | Loss on 100 Sucrose during Evaporation in the Vessels. | Loss on 100 Beet. |
|---|---|---|---|---|---|---|
| Juice heater.. .. | 6½ | 120·8 | 10 | 0·330 | 0·0358 | — |
| First vessel ... | 9 | 106·2 | 15 | 0·120 | 0·0180 | — |
| Second ,, .. .. | 14 | 86·1 | 25 | 0·055 | 0·0128 | — |
| Third ,, .... | 27 | 67·7 | 50 | 0·010 | 0·0045 | — |
| | 56½ | | | | 0·0711 | 0·0092 |

Provided the dimensions of the vessels are proportional, and the amount of juice kept small, the loss of sugar by decomposition remains under 0·01 per cent. on the raw material, even in the most unfavourable case.

The most remarkable fact resulting from these experiments is that high pressure steam in the first vessel does not cause more loss than low pressure steam. This is due to the fact that up to a temperature of 115° C., sucrose decomposes very slowly. In proportion as the decomposition increases with the rise in temperature, the time during which the juice remains in the vessel becomes shorter, for, owing to the increased transmission of heat at high temperature, the heating surface and therefore the juice content become smaller. Owing to this shortened stay in the vessels, the noxious influence of the higher temperature is neutralized, and therefore temperatures of some degress above 100° may safely be employed in the first vessels or in the juice heaters.

That this is true is seen in the Kestner evaporator where the juice to be concentrated only remains for one or two minutes in the tubes, but at a steam temperature of 130° C. in the first vessel and of 110° C. in the second, without any notable charring or decomposition of the sugar or other constituents of the juice.

Besides the double, triple, and quadruple-effets already mentioned, horizontal vessels are also used in the system called "Yaryan," mainly based on the same principle. They have this advantage, that the juice flows rapidly through tubes of small diameter, and is therefore concentrated in a few minutes; but to this end the tubes must be carefully kept clean, and this causes so much trouble that the apparatus seems to find no favour with sugar mauufacturers.

**"Yaryan" Apparatus.**

**Mechanical Losses.**

Apart from chemical losses due to decomposition or over-heating, mechanical loss also arises either from the vapours carrying over the juice as a spray (entrainment), or by the juice actually boiling over. In order to avoid such losses of syrup from the last vessel, a "separator" is usually placed between it and the air-pump. This is a cylindrical vessel provided with perforated diaphragms, which reduce the speed of the vapour and compel it to part with the drops of syrup it carries with it. The separated syrup collects on the bottom and flows back to the last vessel by means of a discharge pipe.

**Separator.**

**Sugar present in Condensed Water.**

When the juice boils over in the first vessel the condensed water in the steam drums of the next vessel will contain sugar, which causes corrosion of the boiler plates if the water be used for feeding the boilers. At the high temperature prevailing in the boilers sucrose becomes decomposed into acids, which attack the plates and thus lead to leakages.

Although an unpleasant smell in the steam at once reveals the presence of sugar in the feed-water, it is necessary to test the condensed water for sugar from

**Testing the Condensed Water.** time to time, because this condition need not arise solely from the juice boiling over, but may also be due to leakages in the tubes of the evaporators which allow the juice to pass into the steam drum as soon as the vacuum is temporarily destroyed by some circumstance or other.

The examination of the condensed water can be made by evaporating 100 c.c. to a volume of 10 c.c., boiling it for a few moments with a few drops of hydrochloric acid, then neutralizing with caustic soda, and heating it again after addition of a little Fehling's solution, in which case a precipitate of red cuprous oxide reveals the presence of sugar. The reaction with an alcoholic $\alpha$-naphthol solution (10 per cent.), of which 1 c.c. with 3 c.c. of sulphuric acid and 1 c.c. of condensed water assumes a blue coloration in case sugar is present, is too sensitive a test, as it detects traces of sugar which are practically insignificant.

From time to time it is also necessary to test the reaction of the condensed water, as it sometimes becomes acid. When sulphitation is carried too far and

**Acid Condensed Water.** gives rise to bisulphites, these decompose in the evaporating vessel, forming normal sulphite and sulphurous acid, which latter is carried over by the vapours, and is thus found in the feed-water for the boilers. Acid water attacks the boiler tubes and plates, causing them to leak.

**Sulphur.** The writer once found sulphur in the condensed water, this being probably due to reduction of the sulphurous acid by the metal of the steam drums.

It often occurs that although the water is free from sugar and acids, it assumes a red coloration in the boilers and primes violently. This is

**Red Colour of the Boiler Water.** due to the water being too soft and pure, and rusting the boiler plates, and can be efficiently remedied by feeding the boilers with calcareous water for a few days, so that the metal becomes covered with a thin scale, which prevents it from being subsequently attacked by the soft water.

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## II.—Incrustations in Evaporators.

Owing to the concentration of the cane juice, various substances which had been dissolved in the thin juice are separated in an insoluble state; they partly

**Nature of the Incrustations.** remain in the juice, rendering it turbid, and are partly deposited as a hard crust on the tubes, chiefly on those of the last vessel, and interfere with the conduction of heat, and hence with the evaporation. It is evident that such an insulating coating on the tubes interferes with the evaporation, and therefore decreases the capacity of the plant. It is therefore desirable, first to avoid the formation of scale as far as possible; and, second, to remove the scale before it becomes so thick as to delay the evaporation.

**Formation of Scale Unavoidable.**

As even the clearest and best clarified juice is certain to deposit constituents, which are insoluble in the concentrated syrup, it is impossible to prevent the formation of scale, so that the only course is to remove it as frequently as possible.

The composition of the scale depends, first, on the nature of the clarification and, second, on the degree of concentration, in other words, on the number of the vessel in the multiple-effet.

**Composition of Scale at different Stages of Concentration.**

The scale in the first vessel chiefly contains constituents which were present in the juice in the state of colloids or in suspension, while that in the other vessels consists principally of substances which were dissolved in the diluted juice, but became insoluble during concentration, either because of the diminished quantity of solvent or by their being less soluble in concentrated sugar solutions. The first vessel, therefore, does not get much scaled when the methods of clarification permit of filtration of the whole of the juice and therefore remove the colloid impurities. On the other hand, the first vessel becomes heavily incrustated when the juice is not filtered but merely decanted or syphoned off. Moreover, when dead or over-ripe canes are being crushed, yielding juice containing much suspended cane fibre, the first vessel may thereby become choked, the fibre being converted into hard masses by the inorganic suspended matter deposited upon it.

**Deposit of Cane Fibre.**

An analysis of such a deposit, although not strictly an incrustation, gave the following figures calculated on dry substance.

| | Per cent. |
|---|---|
| Cane fibre .. .. .. .. .. .. .. .. .. .. .. | 22·0 |
| Silica .. .. .. .. .. .. .. .. .. .. .. .. .. | 44·4 |
| Calcium carbonate and phosphate .. .. .. .. .. | 33·6 |
| | 100 |

In factories working with the defecation process and where, therefore, the greater part of the juice is evaporated without being filtered, the first vessel of the multiple-effet scales much more than in factories where the juice is clarified by carbonatation and consequently is deprived of all colloid matters by filtration.

In the incrustations from the former juice we find calcium phosphate, the quantity of which decreases as evaporation proceeds; also silica, which on the contrary, increases as evaporation proceeds. In the last vessel the scale contains calcium oxalate.

In the case of carbonated juices, the first vessel contains a little calcium carbonate, probably formed by decomposition of bicarbonate and also some silica. In the other vessels the silica-content increases, and in the last calcium oxalate prevails.

Some analyses of incrustations met with in practice follow here, the results being calculated on dry substance, and free from traces of metal from the tubes.

DEFECATION.

| Constituents. | I. Defecation. | | | | II. Defecation. | | | | III. Defecation and Sulphitation. | | | IV. Defecation and Sulphitation. | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | I. | II. | III. | IV. | I. | II. | III. | IV. | I. | II. | III. | I. | II. | III. | IV. |
| Loss on ignition .. | 22·28 | 25·97 | 31·62 | 39·91 | 29·10 | 23·97 | 33·15 | 35·50 | 24·35 | 30·63 | 33·58 | 19·80 | 20·40 | 21·36 | 20·11 |
| Silica .. .. .... | 5·64 | 14·26 | 41·75 | 18·45 | 2·08 | 1·75 | 12·35 | 24·80 | 2·14 | 2·83 | 40·32 | 4·30 | 13·26 | 31·66 | 57·27 |
| Phosphoric acid .. | 29·25 | 22·12 | 9·83 | 2·70 | 21·87 | 25·40 | 6·12 | 0·97 | 23·12 | 21·65 | 7·51 | 22·30 | 22·00 | 10·34 | 0·36 |
| Sulphuric acid ... | 1·90 | 2·31 | 0·45 | 0·26 | 4·84 | 5·87 | 1·10 | 0·94 | 7·92 | 5·48 | 2·36 | 2·01 | 1·29 | 0·84 | 0·41 |
| Sulphurous acid .. | — | — | — | — | — | — | — | — | 0·50 | 0·36 | — | — | — | — | — |
| Carbonic acid .... | 0·57 | 0·55 | 0·38 | 11·50 | 2·12 | 5·17 | 1·84 | 1·35 | 0·25 | 0·47 | — | 4·11 | 3·37 | 3·62 | 3·56 |
| Oxalic acid .. .. | — | — | 1·37 | 5·00 | — | 1·10 | 26·94 | 24·61 | — | — | 2·77 | — | — | — | — |
| Iron oxide .. .... | 1·47 | 2·20 | 0·76 | 1·69 | 3·50 | 3·10 | 2·84 | 1·50 | 8·11 | 7·44 | 0·51 | 18·79 | 6·16 | 11·55 | 6·50 |
| Alumina .. .. .. | 0·30 | 0·39 | 0·13 | 0·81 | 0·31 | — | 0·12 | 0·70 | — | — | — | — | — | — | — |
| Lime .. .. .... | 39·13 | 31·96 | 13·97 | 23·42 | 30·17 | 31·83 | 30·00 | 18·79 | 31·90 | 29·35 | 13·09 | 29·33 | 32·63 | 21·70 | 15·12 |

CARBONATATION.

| Constituents. | V. Single Carbonatation. | | | VI. Double Carbonatation. | | | VII. Double Carbonatation and Sulphitation. | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | | Well conducted. | | | Badly conducted. | | | |
| | I. | II. | III. | I. | II. | III. | I. | II. | III. | I. | II. | III. | IV. |
| Loss on ignition .. .. | 34·58 | 40·63 | 42·25 | 9·65 | 15·33 | 30·76 | 38·04 | 47·72 | 44·84 | 27·66 | 32·67 | 25·30 | 39·74 |
| Silica .. .. .. .... | 24·91 | 38·99 | 14·22 | 12·03 | 42·09 | 42·40 | 14·38 | 9·11 | 9·28 | 1·20 | 2·80 | 2·00 | 2·80 |
| Phosphoric acid .. .. | 0·42 | — | — | 0·61 | 0·18 | — | — | — | — | 0·52 | 0·20 | — | — |
| Sulphuric acid .. .... | 0·38 | — | — | 1·13 | 0·81 | 0·27 | 1·78 | 1·12 | 0·50 | 0·61 | 0·32 | 0·10 | — |
| Sulphurous acid .. .. | — | — | — | — | — | — | — | — | — | 32·05 | 23·73 | 28·00 | 2·19 |
| Carbonic acid .. .... | 8·04 | 0·52 | 0·44 | 27·19 | 10·03 | 5·56 | 9·74 | 5·29 | 0·97 | 1·10 | 1·00 | 2·20 | 2·10 |
| Oxalic acid .. .. .. | — | 4·65 | 30·01 | — | 0·87 | 6·94 | — | 4·32 | 31·21 | — | 9·80 | 11·45 | 41·90 |
| Iron oxide .. .. .... | 16·32 | 11·71 | 4·72 | 4·54 | 5·28 | 3·75 | 17·95 | 18·82 | 7·55 | 2·42 | 2·30 | 1·42 | 0·63 |
| Alumina .. .. .. .. | 0·86 | 0·44 | — | 6·50 | 10·09 | 1·32 | — | — | — | — | — | — | — |
| Lime .. .. .. .... | 12·81 | 4·31 | 21·47 | 38·52 | 16·27 | 12·30 | 13·67 | 17·21 | 22·90 | 30·91 | 29·06 | 34·60 | 30·91 |

The loss on ignition is the measure of the organic substance, but includes the water of crystallization of the gypsum. The sum of the constituents sometimes exceeds 100 per cent., because the weight of carbonic acid liberated from the calcium oxalate on combustion is recorded under the figure for loss on ignition. In addition to oxalic acid, the organic constituents of the scales include gummy and albuminoid substances which are deposited together with other gelatinous bodies, bagasse fibres already mentioned, and a considerable proportion of

caramel, the latter being due to overheating of the juice which penetrates the incrustations and becomes partly charred.

Iron oxide is formed by the solvent action of the juice on the iron plates of the steam chamber; the other constituents, being derived from the juice, some of which were dissolved therein, whilst others occurred in the state of colloids. Among these latter, calcium phosphate occupies the most prominent place, and this explains why the scale in the first vessel chiefly consists of calcium phosphate (with defecated juice), whilst this constituent is almost always absent from the scale formed from carbonatation juice which is filtered previous to evaporation.

**Origin of the different Constituents of the Scales.**

It is true that calcium carbonate, sulphite, and oxalate (found in the scale) are insoluble in water, but they dissolve to some extent in sugar solutions, the solubility depending on the concentration, temperature, and reaction of the sugar solutions.

Calcium carbonate is only slightly soluble in sugar solutions, and when this substance occurs in appreciable proportion in the scale, it must either have been formed from quicklime by absorption of carbonic acid in the defecation process, or have been dissolved in the form of bicarbonate during the second saturation of the carbonatation process, and subsequently decomposed into normal carbonate and carbonic acid during evaporation, and, in consequence, only found in the scale from the first vessels.

**Calcium Carbonate.**

Calcium sulphite is much less soluble in sugar solutions at high than at low temperatures, and is also much less soluble in neutral than in alkaline and acid solution. The reason why this substance is sometimes found in scale, and in large quantity, is not the solubility of the normal sulphite, but that of the more soluble bisulphite. We have seen in former chapters that normal sulphites give an alkaline reaction to litmus paper and if sulphitation is pushed so far that the juice (containing calcium sulphite in suspension) appears neutral or even acid, a considerable amount of bisulphite is formed and dissolved.

**Calcium Sulphite.**

During evaporation, this bisulphite decomposes with the formation of sulphurous acid which escapes as vapour, and the normal sulphite which remains in the juice and is deposited on the tubes of the evaporating vessels. Consequently the largest proportion is found in the scales of the first vessel, where the bisulphite is decomposed; the normal sulphite being so insoluble that sufficient is not left in solution to reappear in the scale formed in the other vessels when the concentration increases by evaporation of the solvent. It is evident that when care is taken during sulphitation to keep the juice faintly alkaline to phenolphthalein paper, no bisulphite can possibly be formed and no sulphite will be found in the incrustations. This is shown in No. VII. of the table on page 205

where in the well-conducted instance after carbonatation and sulphitation, no calcium sulphite was detected in the scale; whereas, in the badly conducted case where sulphitation had been pushed too far and even caused an acid reaction on litmus paper, so much bisulphite was dissolved that the incrustation in the first vessel consisted almost entirely of sulphite. As an equivalent quantity of sulphurous acid passed over into the condensed water, the latter became acid and had a detrimental effect on the boiler plates.

According to Geese* the solubility of calcium sulphite in a 10 per cent. sucrose solution, at the different temperatures, is as follows:—

| 20° | 30° | 40° | 50° | 60° | 70° | 80° | 90° | 100° |
|---|---|---|---|---|---|---|---|---|
| 0·183 | 0·144 | 0·14 | 0·075 | 0·035 | 0·033 | 0·012 | 0·010 | 0·006 per cent. |

The solubility is still less in alkaline juices, especially at high temperatures, so that calcium sulphite can only occur in scale when present in the juice as bisulphite and after being decomposed into normal sulphite and sulphurous acid.

**Calcium Oxalate.**

Calcium oxalate is soluble in sucrose solutions, the solubility increasing in inverse ratio to the temperature and (within certain limits) to the concentration of the solution. A 25 per cent. sucrose solution rendered strongly alkaline dissolves 0·05 per cent. of calcium oxalate, but this figure decreases greatly at higher concentrations and smaller alkalinities. There are, therefore, three factors which render calcium oxalate insoluble during evaporation, viz., the decrease in the amount of solvent, the increased concentration of the sugar solution, and the lowering of the temperature. For a given alkalinity, the solubility of calcium oxalate falls very considerably between the concentration of 30-50 per cent., so that we may expect the heaviest deposit of this substance in the last vessels, which actually occurs. Indeed, the scale of the fourth vessel of a quadruple-effet sometimes consists exclusively of calcium oxalate.

**Gypsum.**

According to Stolle† the solubility of calcium sulphate in sucrose solutions falls in proportion as the concentration and temperature rise. At low temperatures, a sucrose solution dissolves more gypsum than corresponds with its water content, and this is also the case at higher temperatures in low concentrations. But it dissolves less than corresponds with the water content at higher concentrations.

**Calcium Silicate and Aluminate.**

Calcium silicate and aluminate also dissolve in sugar solutions, especially when the solution is strongly alkaline. After long continued heating, lime is withdrawn from these combinations, so that silica and alumina become insoluble and are deposited as scale. Beside the silica occurring in this form, there is no doubt that it is also present as gelatinous silica, suspended in the juice and deposited during evaporation. Otherwise, the fact

* *Zeitschr. Rübenzuckerind.*, 48, 103.

† *Zeitschr, Rübenzuckerind.*, 50, 321.

that acid juices (free from calcium silicate) deposit silica during evaporation could not be explained.

Alumina is sometimes found in scale, although this constituent occurs in but small quantity in cane juice. Being specially noticeable in scale from juice treated by carbonatation, we are justified in concluding that it is derived from the lime used for the clarification, and which sometimes contains as much as 1 per cent. alumina.

**Alumina.**

**Nitrogen.** The nitrogen content of the dry scale is due to coagulated albuminoids, and never amounts to more than 0·20 per cent., and in most cases is less.

The scale in the first vessel sometimes contains a relatively large proportion of cane wax which was suspended in the juice. Some deposits contain so much wax that the sight-glasses of the evaporators become covered with a greasy, dark-coloured film. Owing to its low specific gravity, such a film floats on the surface of the juice, and therefore wax is not a notable constituent of hard scale.

**Cane Wax.**

The following is a typical analysis:—

| | |
|---|---|
| Calcium phosphate .. .. .. .. .. .. .. .. .. | 48·7 |
| Cane wax .. .. .. .. .. .. .. .. .. .. .... | 40·2 |
| Silica .. .. .. .. .. .. .. .. .. .. .. .. | 5·7 |
| Undetermined organic matter .. .. .. .. .... | 5·4 |
| | 100· |

It goes without saying that thorough subsidation, or, if possible, filtration of the juice will help to prevent incrustation by suspended matter, and further that a neutral juice can dissolve less lime salt than an acid or an alkaline one, and, therefore, will be less liable to deposit scale.

As juices are very seldom exactly neutral, they all deposit incrustations, and all that can be done is to restrict their formation as much as possible, and to remove them as soon as they have formed.

Some ten years ago, iron spirals were suspended in the tubes of the evaporating vessels, with the object of inducing the scale to deposit on these, rather than on the tubes. About the same time wooden staves ($\frac{1}{3}$ inch less in diameter than the tubes) were similarly used, with the additional advantage of diminishing the volume of juice, and thus increasing the heating surface. The rough surface of the staves was also more favourable for the deposit of scale than the smooth surfaces of the brass tubes. Though this novelty was at first regarded very favourably, the staves have since disappeared.

**Removal of the Scales.**

Such appliances do not obviate the cleaning of the vessels from time to time. In countries where the mill stops on Sundays, that day can be utilized for this

purpose, while in places where the work continues all seven days of the week without rest, it is well to clean and scrape the evaporators say once a fortnight. The practice, sometimes followed, of continuing evaporation without stoppage until the evaporators are too much scaled, is to be condemned, because the capacity of the plant will be considerably reduced during the last days, and also because the layer of scale has then become so thick that it costs a great deal of labour and time to remove it.

**Boiling with Caustic Soda.**

The best mode of cleaning is to boil caustic soda solution in the vessels, with open valves and with live steam in the steam drum, so that the liquid boils at ordinary atmospheric pressure, otherwise the temperature is not sufficiently high. After some six or more hours the caustic soda solution is let out, the vessels are washed a couple of times with water, and the scale scraped off with scraping irons. This last operation, being somewhat rough, is not without danger to the brass tubes, as appears from the rather large quantity of brass splinters which were found in every sample of scale examined by the author. Formerly, the vessels in which the soda solution had been boiled were boiled again with dilute hydrochloric acid after the soda had been let off, and the vessels washed. This was to dissolve the already loosened scale, but the practice is now discontinued. It is true that most of the incrustations dissolve in concentrated hydrochloric acid, but this should not be applied, since it attacks the iron vessels and results in leakages. Diluted hydrochloric acid dissolves the calcium carbonate found in scale in the first vessels treating carbonatation juice, but it fails to attack the crystallized oxalate and the silica, so that the action of this reagent is very restricted. It also has the disadvantage of attacking the unscaled portions of the iron plate, with the formation of hydrogen, which forms an explosive mixture with air. No workmen should, therefore, enter the vessels with a light after boiling with hydrochloric acid before the latter have been thoroughly washed out with water, and the air drawn off by the air pump. Hydrochloric acid does not dissolve much of the scale, neither does caustic soda, but this last reagent loosens it, so that it may be more easily scraped off. Some silica becomes dissolved and calcium oxalate is decomposed, but otherwise the composition of a scale is not much changed after being boiled with caustic soda.

**Composition of Scale before and after treatment with Caustic Soda.**

The analytical figures given below relate to the washed and dried scales after treatment with caustic soda, and are from the same factories as the sample numbers I. and V., on page 205, which were analysed before boiling with caustic soda. From the insignificant difference in composition before and after boiling with caustic soda, we see that this reagent has a loosening effect rather than a solvent action.

| | Defecation Scale after treatment with Caustic Soda. | | | | Carbonatation Scale after treatment with Caustic Soda. | | |
|---|---|---|---|---|---|---|---|
| | I. | II. | III. | IV. | I. | II. | III. |
| Loss on ignition .. | 26·20 | 27·60 | 40·01 | 35·30 | 24·92 | 33·55 | 27·00 |
| Silica .. .. .... | 7·28 | 8·43 | 22·49 | 39·01 | 44·19 | 42·98 | 35·85 |
| Phosphoric acid.. .. | 20·91 | 19·85 | 1·61 | 0·37 | 0·45 | 0·26 | — |
| Sulphuric acid ... | 4·87 | 3·38 | 0·43 | — | 0·60 | 0·31 | — |
| Sulphurous acid.. .. | — | — | — | — | — | — | — |
| Carbonic acid.. .... | 3·97 | 4·08 | 3·06 | 3·81 | 2·68 | 2·74 | 3·46 |
| Oxalic acid.. .. .. | — | — | 20·49 | 12·60 | — | — | 10·97 |
| Iron oxide .. .... | 2·24 | 3·47 | 0·69 | 0·55 | 20·11 | 12·14 | 14·54 |
| Alumina .. .. .. | — | — | — | — | 1·22 | 1·41 | 0·48 |
| Lime.. .. .. ... | 34·29 | 31·28 | 20·15 | 13·96 | 5·64 | 5·69 | 12·01 |

**Removal of Scale by fermenting Diluted Molasses.**

A sure but slow means of cleansing the vessels after the close of the grinding season is to fill them with diluted molasses (6-8° Brix) and allow this to ferment. The evolution of gas within the scale itself loosens the particles of which it consists, and after some three weeks the fermented wash is run off, and the scale may then be brushed away without scraping. As this method requires time, it can not, of course, be employed during the grinding season, so that boiling with caustic soda and scraping is then the only remedy.

**Deposits in the Steam Chambers of the First Vessels.**

The deposits so far mentioned are derived from the juice, but another troublesome deposit occurs in the steam chamber of the first vessel, which likewise retards the transmission of heat, and evaporation. The steam used for heating the first vessel may contain small quantities of lubricating oils, which condense when coming in contact with the cold tubes and which, in combination with iron or copper oxides, graphite, and similar substances, form a greasy, non-conducting layer which causes much trouble. The author has examined several such deposits, and his analyses, calculated on dry substance, are recorded here. The first sample dates back to 1892 and must have been gradually forming during the ten preceding years. The organic matter chiefly consisted of fatty acids, while in the second and third samples, it consisted of mineral lubricating oils.

| Constituents. | I. | II. | III. |
|---|---|---|---|
| Oil .. .. .. .. .. .. | 28·4 (fatty acids) | 28·50 | 13·9 |
| Iron oxide.. .. .. .... | 39·4 | 58·80 | 82·6 |
| Copper oxide.. .. .. .. | 30·0 | 2·50 | — |
| Lead oxide .. .. .... | 1·0 | 6·20 | 2·3 |
| Graphite, &c. .. .. .. | 1·2 | 4·0 | 1·2 |

Such a layer is sometimes, but not always, found in the steam chambers; and as its presence cannot be detected from outside, it is advisable to inspect the steam chamber every year, and to remove the deposit if present. Mineral oils do not dissolve in soda or in acids and therefore boiling either with acids or alkalies is of no use. It can be removed by dissolving it in petrol (gasoline) or benzine. The fatty deposit is thereby softened and flows off as a greasy mud; after which the spirit is run off and the steam chamber washed free from solvent with water. This method is rather dangerous because of the inflammable vapours escaping from the spirit. It should therefore never be attempted during the grinding season, but only when factory operations have come to a complete standstill.

**Removal of the Greasy Deposit.**

If this method be considered too dangerous, the only other remedy is to remove all the tubes and scrape them carefully.

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## III.—Clarification of the Syrup.

Though the greater proportion of the substances which become insoluble during evaporation deposit on the walls and tubes of the vessels, yet a part remains in the syrup rendering it turbid.

The composition of these floating and suspending substances is similar to that of scale, as can be gleaned from an analysis of a deposit from syrup tanks in a factory working with the defecation process.

**Composition of Deposit from a Syrup Tank.**

| | |
|---|---|
| Loss on ignition | 34·33 |
| Silica | 26·95 |
| Phosphoric acid | 12·25 |
| Sulphuric acid | 2·03 |
| Carbonic acid | 0·23 |
| Iron oxide and alumina | 3·58 |
| Lime | 19·45 |
| Undetermined | 1·18 |
| | 100 |

This sample closely resembles the average of the scales from the different evaporating vessels, and consists of the same elements. It is very important to remove these impurities, because they become enveloped in the sugar crystals during the growth of the latter and impart to them a dark tint which cannot be removed by washing.

It is not feasible to remove these impurities by filtration through cloth if the syrup has been obtained from defecated juice, since the gummy constituents clog the filter cloths or other filtering media, consequently one is compelled to allow the syrup to subside.

**Removal of same.**

Sand filters which were greeted with enthusiasm a couple of years ago, do not seem to have fulfilled early promises, since they have not yet come into general use for syrup. It seems that the very large quantity of water required to wash out the syrup from the sand when cleaning is the chief obstacle to their employment for this purpose.

**Filtration.**

Syrup from juice clarified by the carbonatation process, after being heated to a favourable temperature, easily passes through any kind of juice filter and yields a limpid and bright syrup. At times, however, it does not filter clear and attempts have been made to remove this cloudiness by adding 0·01 per cent. of Soxhlet's filtering medium to the syrup before filtration. This medium consists of one part of infusorial earth and two parts of woodpulp; it is stirred with the syrup and the mixture passed through filter-presses. Although this material gave satisfactory results in one factory, it has not gone further than the experimental stage.

The syrup from defecated juice cannot be filtered, and can only be clarified by subsidation. The best method is to boil the syrup in eliminating pans after it leaves the evaporating plant, neutralizing it with lime or soda if necessary, or, if too alkaline, with phosphoric acid. The chemicals mentioned in the previous chapter, such as potassium ferrocyanide, &c., may also be added with advantage. Sulphitation may be combined with this elimination, but a stirring apparatus is then necessary in order to bring the gases into contact with the syrup. It is advisable to keep the temperature of the syrup below 70° C. in order to prevent inversion during sulphitation.

**Subsidation.**

If the precipitate subsides slowly, a little phosphoric acid and lime, soda, or sodium phosphate, may be added during elimination, but great care must be taken not to use too much of these, or the precipitate might become too thick. If possible, the syrup is then filtered. If not, it is run into settling tanks similar to those described in the chapter on clarification. A clear supernatant syrup then separates from a layer of mud, the thickness of which depends on the quantity and the flocculency of the precipitate.

It is advisable to decant the subsided syrup as soon as possible, and not to leave it in the tank too long. According to Herzfeld's tables, mentioned on another page, at a temperature of 90-95° C. such a syrup loses 0·0392 on 100 sucrose per hour, so that during the six hours which syrup generally requires to subside, no less than 0·25 per cent. of the sucrose may become decomposed.

As soon as the syrup has settled, it is drawn off from the precipitate and conveyed to the suction tank of the vacuum pan, while the precipitate itself is dealt with in various ways. Some manufacturers mix it with the molasses coming from the centrifugals, and boil them together to second sugars. There could not be any objection to this plan if the second sugars always

**Treatment of the Syrup Scums.**

found a market, for the fine particles of the precipitate would be got rid of in the crystals of the second sugars. In many cases the second sugars are re-melted in the juice, and the impurities, previously eliminated with care, are thereby returned to the juice. Moreover the practice of returning molasses to the pan has suppressed the second sugars, so that the mixing of the syrup scums with first molasses has become impossible. In other factories, the syrup scums are run into the scum-tank of the mill juice, which proceeding has the drawback of increasing the work of the scum filters, and thus diminishing the capacity at that stage. Further, this scum is very slimy, and the particles are excessively fine, so that they choke up the pores of the filter-cloths. Finally, these scums contain a highly concentrated sugar solution, and as even the driest press cakes contain about 50 per cent. of juice it is not an economical proceeding, since we are here dealing with concentrated juice or syrup, in which a considerable quantity of sugar becomes lost. The best way is to pump the syrup scums into the juice in the defecation pans; the syrup mixes with the great volume of mill juice without any visible increase of concentration, and again undergoes all the operations of clarification and settling, while the fine precipitate is carried down by the flocculent scums of the mill juice, subsides with them, and does not give rise to any increase in the quantity of juice to be filtered by the presses. Moreover, the higher sugar content of this juice is so trifling, that the loss of sugar in the filter-press cakes is not perceptibly increased.

**Sourness of Syrup by Fermentation.**

Sometimes, juice which is neutral after defecation, yields a very acid syrup, but in this case no attempt should be made to remedy the defect by using more lime in tempering, for, strange as it may appear, this is more apt to increase the acidity of the syrup than to diminish it. The only thing is to neutralize the quantity of syrup in hand, and immediately seek the cause of this acidity. In every case where a neutral clarified juice becomes very acid during manufacture, this is generally due to some ferment or other which attacks sucrose and forms acid from it. As a rule, lime salts favour these fermentations, hence an excessive use of lime promotes the deterioration. This can be remedied by keeping the amount of lime as low as possible, and by great care in cleaning all pipes, tanks, and pumps, and even disinfecting them with a one per cent. solution of ammonium fluoride. It is of course better to prevent such infection by washing out the gutters and settling tanks daily.

Juices containing much glucose show a rapid decrease in alkalinity which does not lead to sourness as is the case with fermentation.

## IV.—Boiling.

**Principle.** During the boiling process the concentration of the clarified syrup is continued up to the point where there is not sufficient water for the sugar to remain dissolved, and it becomes partially crystallized.

The rapidity with which sugar crystallizes out from a solution depends, other circumstances remaining the same, on the purity of the solution, *i.e.*, on whether much or little foreign substance is present along with the sugar.

**Rapidity of Crystallization.** When the sugar solution is fairly pure, crystallization takes place as soon as the concentration has become too great for all the sugar to remain in solution: when an impure juice such as molasses is evaporated, crystallization takes place only very slowly, and no crystals are visible, even when the concentration is much greater than corresponds with the solubility of sucrose.

The hot concentrated liquid is allowed to cool, and after some time sucrose crystallizes out, partly because it is less soluble in cold water than in hot, but chiefly because the supersaturated state slowly gives place to the normal state of solubility.

**Different Ways of Boiling.** Boiling may therefore be performed in two ways, one of which is followed when boiling syrup, and the second when boiling molasses, but as will be shown later on this distinction is not strictly observed.

Pure juices are concentrated at not too high a temperature until the sucrose commences to crystallize in the pan, after which the crystals grow slowly as evaporation continues and fresh supplies of syrup are added, until ultimately the whole is transformed into a stiff *magma* (masse cuite), consisting of a large quantity of crystals and only a little liquid (molasses). This method is called "boiling to grain," and can only be applied to pure syrups. If the solution is too impure for this purpose, it is concentrated at moderate temperatures to such a degree that the concentrated syrup or molasses only deposits sugar on cooling, this mode of working being termed "boiling smooth." The consistency of the liquid being such that a sample can be drawn out in the form of a thread, the liquid is said to be boiled to "string proof."

Boiling is always performed *in vacuo* for the reason given under the heading "Evaporation," and for the further reasons that the temperature at which the heavy material parts with its water under atmospheric pressure is still more elevated; and circulation being less perfect, the risk of local

**Boiling in vacuo.**

over-heating and consequent decomposition and coloration of the masse cuite increases.

**Vacuum Pans.** The old vacuum pans were made of copper, but owing to the demand for pans of large capacity they are now almost exclusively made of iron, so that copper pans provided with steam jackets are now the exception. The iron vacuum pans are in many cases lined with copper plates in order to prevent coloration of the sugar by the solution of iron in the acid syrups and molasses.

**Heating.** In most cases pans are heated by means of copper coils, to which either direct or exhaust steam is admitted.

**Steam-jackets.** As already mentioned, the earliest pans had steam-jackets in addition to coils, but these were subsequently dispensed with in order to secure a wide discharge opening, and thus to economize time when discharging the pan. The advantages of a steam-jacket are that the heating surface of the pan is increased by several square feet, and the masse cuite in the pan is less exposed to the cooling influence of the external atmosphere. Consequently, steam-jackets have been adopted in many modern pans, even the discharge door being heated in this manner.

**Steam Chambers.** Instead of coils and steam-jackets, many pans have steam chambers, similar to those used in multiple-effet evaporators, but with tubes of much larger diameter to permit the semi-solid masse cuite to circulate. In many cases the circulation is further promoted by a screw revolving in a large central tube whereby the boiling mass is raised in the centre, and thus induced to flow downwards at the sides.

**Circulation.** It is of great importance that the contents of the pan be thoroughly mixed; first, in order to prevent local overheating of the masse cuite where it is in contact with the hot coils; and, secondly, in order to secure crystals of uniform size. Circulation is especially necessary when molasses (previously separated from crystals) are used for diluting the masse cuite in the pan, otherwise the concentrated masse cuite settles to the bottom of the pan, and does not mix with the added molasses. Under such conditions, samples taken with the proof stick give false indications concerning the real state of the contents of the pan. Even when boiling syrup masse cuite, the bottom discharge is often choked by a hard mass of sugar which has been accumulating for a considerable time, and has interfered with the circulation. In order to prevent this, and, in general, to promote circulation, many pans are provided with a perforated copper coil through which dry low pressure steam is blown through the boiling mass, so as to mix it, the vacuum not being perceptibly lowered. The perforations should be made at the bottom of the coil, in order to prevent them being

choked with sugar crystals when the pan is discharged. A second device for promoting circulation is to introduce the syrup and molasses through a bent pipe extending nearly to the bottom of the pan, so that the thin liquids are compelled to force their way upwards through the masse cuite and so become thoroughly mixed with it, which is not the case if the syrup is introduced at the top of the masse cuite. The same end is sometimes gained by pumping the syrup and molasses through a perforated copper coil at the bottom of the pan. To prevent choking this coil is likewise perforated on its under side.

In order to examine the contents of the pan without disturbing the vacuum, samples are withdrawn from time to time by means of the apparatus now to be described. A copper tube is fixed in an inclined position within the pan in such a manner that the upper extremity projects through the side of the pan while the lower extremity terminates at the centre of the pan and, consequently, is immersed in the boiling masse cuite. The upper extremity of this tube is open but the lower extremity is closed by a valve of special construction. When a sample is to be withdrawn from the pan, this valve is opened by means of a long brass rod which is thrust down the tube and then turned as one turns a key in a lock. This rod, called the "proof-stick," is provided at its lower end with a plug by means of which the above-mentioned valve is opened, and also with a small cavity which thereby becomes filled with the boiling material. During this operation, the upper opening in the tube is kept air-tight by the closely fitting rod or "proof-stick." The latter is then twisted back to its former position, thereby locking the valve, and then withdrawn from the tube carrying with it the small sample of masse cuite which fills the cavity.

**Proof-stick.**

The proof-stick should be of sufficient length that samples may be withdrawn from the bottom part of the pan, and the cavity sufficiently large to withdraw a fairly large sample at each test.

**Fittings of the Pans.**

Chemicals are drawn into the pan through an iron pipe fitted at the side, whilst a cock near the top of the pan (called the butter-cock) may be used for introducing colouring agents, such as ultra-marine, &c.

The pan is connected with an air-pump and injector for removing and condensing the vapours, and the resulting vacuum permits of syrup, molasses, chemicals, &c., being sucked into the pan without being pumped as is the case in open evaporators. Between the pan and the air-pumps is fixed a kind of separator called the "save-all," which keeps back the spray rising from the boiling liquid and returns it to the pan.

When boiling to grain, syrup is drawn into the pan as soon as the vacuum permits, and when the bottom coil is covered, exhaust steam is turned on.

**Boiling to Grain.** As the level of the liquid rises by the constant inflow of syrup, steam is similarly admitted to the second and, if necessary, also to the third coil, the proper charge of syrup being found by experience ; the supply of syrup is then stopped and concentration commences. The formation of grain commences at about 80° Brix, and is accelerated in one of two ways. The syrup is either concentrated until it begins to crystallize, or additional syrup is suddenly added to the already supersaturated syrup, causing such an agitation throughout the mass that the sugar (present in a supersaturated state) is deposited as fine glittering crystals. This addition of dilute syrup suddenly reduces the boiling point and causes a brisk ebullition and thereby agitation of the whole liquid which promotes crystallization. The temperature in the pan is usually 50° C. (122° F.) and the vacuum 28 inches (71 cm.).

When the pan is heated by a steam chamber or calender instead of by coils, it is necessary first to draw in enough syrup to immerse the whole heating surface, after which the concentration of the syrup up to the graining point is carried out as before. But it may be necessary to start with less syrup than will completely cover the steam chamber, so that coils are to be preferred unless it is practicable to transfer part of the contents of one pan to another, after the grain has been formed, and to continue boiling what is left.

**Determination of the Graining Point.** Although every pan is provided with thermometer and vacuum gauges, and the boiling can be perfectly regulated by means of these instruments, most native pan boilers prefer to judge the concentration of the syrup from the appearance of the drops of liquid spattering against the sight glasses.

Although this test seems very crude and uncertain, experienced pan boilers know pretty well what to expect of the juice with which they have to deal, and when the syrup does not grain at the point they are accustomed to, they will go on concentrating before attempting "to grain" by the sudden addition of fresh syrup.

As cane juice grains very easily, the artifices employed in the beet sugar industry to promote crystallization are superfluous. On the contrary, it is sometimes difficult to prevent premature formation of grain.

**Brasmoscope.** Some years ago, Curin* constructed an instrument which he called a "brasmoscope," and which indicated the density of the saccharine liquid boiling in vacuo. He started from the observation, that between the limits of 57½-48½ cm. vacuum (22·6-19·1 inches), and 65-74° C. (149-165·2° F.) every cm. difference in vacuum (mercury pressure) corresponds with a difference of one degree centigrade in the boiling point of pure water.

* *Oest. Ung. Zeitschr. f. Zuckerind.*, 1894, 756.

Therefore, if a barometer divided in cm. and a centigrade thermometer be together immersed in water boiling in vacuo, the mercury will rise the same amount in both instruments within the limits mentioned above. This is shown by the following table of the tension of water vapour according to Regnault and Bloch.

| Temperature in °C. | Tension of the Vapour in cm. Mercury. | Vacuum in cm. Mercury. | Temperature in °C. | Tension of the Vapour in cm. Mercury. | Vacuum in cm. Mercury. |
|---|---|---|---|---|---|
| 64 | 17·89 | 58·11 | 70 | 23·33 | 52·67 |
| 65 | 18·71 | 57·29 | 71 | 24·36 | 51·64 |
| 66 | 19·57 | 56·43 | 72 | 25·43 | 50·57 |
| 67 | 20·46 | 55·54 | 73 | 26·54 | 49·46 |
| 68 | 21·38 | 54·62 | 74 | 27·69 | 48·31 |
| 69 | 22·34 | 53·66 | 75 | 28·88 | 47·12 |

The agreement is very close in the case of boiling water, but less so with syrup, especially when concentrated. But, when once the difference is ascertained empirically, the specific gravity of the boiling syrup can be determined from the readings of the thermometer and vacuum-gauge. In Curin's apparatus, a scale of degrees Brix is calculated for the differences between temperature and vacuum, enabling the density of the boiling liquid to be read off from a sliding scale.

The brasmoscope is only used for syrups or molasses free from grain; when grain is present it only shows the density of the mother liquor in which the crystals float. When the masse cuite contains much grain, the temperature varies so much in different parts of the masse cuite (owing to imperfect circulation) that the readings by no means correspond with the average temperature of the masse cuite.

As the graining point can be determined just as well without special apparatus, the brasmoscope has not been much employed in cane sugar factories, and it is very likely that the refractometer will take its place.

**Composition of Cane Syrups at Graining Point.**

The analyses of a great number of syrups at their graining point are set forth here.

| No. | Brix. | Polarization. | Reducing Sugars. | Water. | Quotient of Purity. | Glucose Ratio. | Sucrose on 100 Water | Sucrose crystallized out on 100 Masse Cuite at 45° C. |
|---|---|---|---|---|---|---|---|---|
| 1 | 79·21 | 71·70 | 3·34 | 21·59 | 90·52 | 4·66 | 332·1 | 19·52 |
| 2 | 80·32 | 72·23 | 3·63 | 20·88 | 89·93 | 5·07 | 345·9 | 22·26 |
| 3 | 79·73 | 69·00 | 5·16 | 21·27 | 89·65 | 6·25 | 325·8 | 17·59 |
| 4 | 77·65 | 70·81 | 3·97 | 23·65 | 89·17 | 5·57 | 299·4 | 13·65 |
| 5 | 78·43 | 71·11 | 3·91 | 22·67 | 88·68 | 5·60 | 326·9 | 16·32 |
| 6 | 80·62 | 71·55 | 3·99 | 20·08 | 88·62 | 5·58 | 356·3 | 23·02 |
| 7 | 81·72 | 71·87 | 5·04 | 19·58 | 87·95 | 7·01 | 367·1 | 24·55 |
| 8 | 78·59 | 68·95 | 4·52 | 22·61 | 87·45 | 7·14 | 304·9 | 14·30 |
| 9 | 83·11 | 72·48 | 3·40 | 17·99 | 87·21 | 4·7 | 402·9 | 29·00 |
| 10 | 79·26 | 69·00 | 5·16 | 21·74 | 87·06 | 7·48 | 317·4 | 16·45 |
| 11 | 78·47 | 67·49 | 4·82 | 22·73 | 87·04 | 7·14 | 297·0 | 12·55 |
| 12 | 83·50 | 72·52 | 4·18 | 17·40 | 86·85 | 5·76 | 417·8 | 30·46 |
| 13 | 78·51 | 68·15 | 5·59 | 22·39 | 86·81 | 8·2 | 304·4 | 14·03 |
| 14 | 79·55 | 67·54 | 5·64 | 21·45 | 84·89 | 8·35 | 314·9 | 15·70 |
| 15 | 79·75 | 66·49 | 6·71 | 21·49 | 83·37 | 10·09 | 309·4 | 14·55 |
| 16 | 75·95 | 63·14 | 7·60 | 25·19 | 83·13 | 12·0 | 250·6 | 5·61 |
| 17 | 79·10 | 65·67 | 3·85 | 21·60 | 83·02 | 5·86 | 304·0 | 13·47 |
| 18 | 81·05 | 66·77 | 3·71 | 19·75 | 82·38 | 5·55 | 338·1 | 19·03 |
| 19 | 84·73 | 69·52 | 4·97 | 17·67 | 82·05 | 7·15 | 393·4 | 24·12 |
| 20 | 85·56 | 70·19 | 4·70 | 15·54 | 82·03 | 6·7 | 451·7 | 32·63 |
| 21 | 81·60 | 66·9 | 8·26 | 19·43 | 82·00 | 12·3 | 344·3 | 19·84 |
| 22 | 78·74 | 64·13 | 4·11 | 22·46 | 81·44 | 6·41 | 285·5 | 9·85 |
| 23 | 83·83 | 68·24 | 5·43 | 19·54 | 81·37 | 8·0 | 354·6 | 21·01 |
| 24 | 80·36 | 65·0 | 7·78 | 20·75 | 80·90 | 12·0 | 313·2 | 14·85 |
| 25 | 82·08 | 66·3 | 7·81 | 18·17 | 80·79 | 11·78 | 364·9 | 22·18 |
| 26 | 85·29 | 68·48 | 5·34 | 17·02 | 80·29 | 7·78 | 402·3 | 27·34 |
| 27 | 80·28 | 64·34 | 8·08 | 20·89 | 80·14 | 12·56 | 308·9 | 13·85 |
| 28 | 83·20 | 66·40 | 8·26 | 18·07 | 80·00 | 12·4 | 367·4 | 22·72 |
| 29 | 77·07 | 59·24 | 8·28 | 26·21 | 76·87 | 13·97 | 226·0 | — |
| 30 | 85·87 | 65·21 | 8·24 | 16·68 | 75·94 | 12·64 | 391·6 | 24·89 |

In this table the sucrose content is assumed to be identical with the polarization, on account of the relatively low percentages of reducing sugar. Further, we have assumed that the solubility of sucrose in the water of the syrup is the same as in pure water* and that the sugar is grained at 45° C. According

* As will be seen later on, the solubility is less in the case of syrup and still less in that of molasses.

to Herzfeld's table on page 4, the solubility of sucrose in water at 45° C. is 71·32 parts of sucrose in 26·68 parts of water, or 241·7 parts of sucrose in 100 parts of water. When calculating from these values the amount of sucrose actually dissolved in the syrup, we can find by difference the amount of sucrose present in a supersaturated form, and which could crystallize out at the given temperature if the syrup were strongly agitated.

**Calculation of the Amount of Sugar Crystallizing out on Graining.**

The quantities of sugar capable of crystallizing differ very considerably, and in Nos. 16 and 29 the graining point was very probably missed and the pan-boiler compelled to concentrate again before obtaining sufficient grain. It is evident that, for a given concentration, the supersaturation is greater in pure than in impure syrups, and therefore the former forms grain more readily than the latter, partly because a pure syrup contains more sucrose per 100 parts of dry substance and partly because this sucrose crystallizes out more easily from pure than from impure solutions.

A few examples taken from the foregoing table will show how the crystallizable sugar varies with the purity of the syrup, although in reality this difference is somewhat compensated for by the lower solubility of sucrose in impure syrups.

| Water. | Quotient of Purity. | Sucrose in super-saturated Solution. | Water. | Quotient of Purity. | Sucrose in super-saturated Solution. |
|---|---|---|---|---|---|
| 19·58 | 87·95 | 24·55 | 21·59 | 90·5 | 19·52 |
| 19·54 | 81·37 | 21·01 | 21·74 | 87·06 | 16·45 |
| 20·88 | 89·93 | 22·26 | 21·60 | 83·02 | 13·47 |
| 20·89 | 80·14 | 13·85 | 22·67 | 88·68 | 16·32 |
| 21·45 | 84·89 | 15·70 | 22·61 | 87·45 | 14·30 |
| 21·49 | 83·37 | 14·55 | 22·73 | 87·40 | 12·55 |

The graining point is, therefore, not only determined by the density, but also by the purity of the syrups, and it is therefore not surprising when an experienced pan boiler sometimes fails to grain a pan of syrup at the first attempt, and only succeeds after a few trials.

Care should be taken, at the outset, to form as large a quantity of crystals (numerically) as should be present in a full-grown state in the finished masse

Boiling. cuite. When making a coarse grained sugar, fewer crystals will be required than when making a fine grained sugar. In the former case it is customary to grain "low down," that is to say, to start with only sufficient syrup to form the required quantity of grain, which is then allowed to grow by constantly drawing in syrup. In this case, we cannot make use of all the steam-coils, because steam may only be admitted to those which are covered by the syrup; consequently, boiling proceeds very slowly. But when making fine grained sugar, we may at once employ the whole heating surface, because the graining is performed "high up," when all the coils are immersed. The pan may, therefore, be filled at once and steam admitted to all the coils, and the whole mass be concentrated to the graining point. But when, owing to evaporation, the volume of syrup diminishes to such an extent that the topmost coils are exposed, steam must be shut off from these. It is evident that when graining high up, the boiling goes on more rapidly than when graining low down, although in this latter case the operation may be much accelerated by graining sufficient syrup in one pan to supply two pans with grain, the grained syrup being then divided between two pans with the same result as if each had been grained "low down." In this way, coarse grained sugar may be obtained in a pan heated by a steam chamber, which is not otherwise feasible.

By spreading a proof-stick sample upon a piece of glass plate, an experienced pan boiler can judge whether the number of crystals in the grained syrup is sufficient for the kind of sugar required. If not, he re-dissolves the crystals by the addition of fresh syrup and increasing the temperature, and tries again after he has concentrated a little further.

By the careful addition of more syrup the grain is allowed to grow regularly, care being taken that the concentration of the liquid (in which the crystals float)

Regular Growth of the Crystals. does not become as high as before graining, since this might give rise to a secondary crystallization (*i.e.*, formation of minute crystals between those already formed). When this happens, it is detected by the turbid appearance of the mother liquor in the samples from the proof-stick,

False Grain. and these crystals, known as "false grain," must be re-dissolved. To this end a large quantity of syrup is drawn into the pan, the injection diminished, and more steam turned on in order to raise the temperature. The fine crystals are thereby dissolved, the original crystals being somewhat diminished in size; but, by causing the temperature to fall slowly, the latter commence to grow again. Syrup is drawn into the pan either in a continuous stream or at intervals until the pan is full. The pan-boiler must not force the concentration too much towards the end of the operation, because if false grain is now formed it can no longer be dissolved owing to imperfect circulation. We are then compelled to leave it in the masse cuite, the consequences of which will be discussed later on.

When the pan is full, the supply of syrup is stopped and the contents concentrated to a Brix of 92-93 (water content = 9-11) usually at a temperature of 65° C. (149° F.) and a vacuum of 730 mm. (28·7 inches). The temperature and vacuum may be read from the gauges, but the exact point at which the concentration should be arrested, *i.e.*, the striking point, has to be found by experience, and is determined by the firmness of a sample (taken by means of the proof-stick), when suddenly cooled in a pail of water.

**Finishing.**

**Discharging the Pan.**

As soon as the masse cuite is ready for discharging, the steam is shut off, the air-pump and injector stopped, and air admitted to the pan by opening a cock in the top of same. The discharge door can then be opened and the masse cuite falls from the pan into suitable receivers.

**Cutting.**

When the grain is very small and a larger grain is wanted, only half the contents of the pan are discharged, and after being again closed fresh syrup is added and boiled so as to permit the crystals to grow larger, a process which bears the name of "cutting."

**Steaming.**

When the pan is empty a certain amount of masse cuite always remains attached to the coils and to the walls of the pan, which, if allowed to remain, would become charred by overheating and produce dark-coloured lumps in the sugar of the next strike. Formerly, those crusts were removed by steaming out the pan, when they became loose and fell through the discharge opening. The steamings were kept separate, so that those from several operations might be cured together, or they were dissolved in the cane juice. But later, the discharge opening of the vacuum pan was frequently connected directly with the crystallizers, rendering the separate collection of the steamings impossible. This difficulty sometimes induced pan boilers to abandon steaming out their pans, with a deleterious effect on the appearance of the sugar. The best remedy is to draw clarified juice into the pan as soon as this is empty and ready to receive a fresh charge of syrup. This juice enters the pan through a pipe of 1 or 2 inches diameter, carrying a perforated nozzle at the top of the pan. The juice falls in a gentle shower against the coils and walls of the pan, detaching the crusts of sugar and dissolving them completely, while a fresh supply of syrup is being concentrated in the pan.

**Composition of a Masse Cuite and of the Molasses contained therein.**

One of a series of analyses of masse cuite and of the molasses occurring therein, between the crystals, is given below. In this example, a pure syrup was concentrated, grained, and boiled, and from time to time samples were taken from the pan and divided into two portions. One of these was analysed, and the other cured in a hand centrifugal to separate the molasses, which were also analysed. The following results indicate the composition of the masse cuite, and of the contained molasses at several stages of the boiling process.

| Nature of the Sample and Stage of the Process. | Dry Substance. | Brix. | Polarization. | Sucrose (Clerget). | Reducing Sugar. | Ash. | Actual Purity. | Apparent Purity. | Temperature. | Vacuum. |
|---|---|---|---|---|---|---|---|---|---|---|
| Concentrated syrup at the graining point.. | 80·43 | 81·61 | 69·0 | 70·21 | 5·20 | 1·80 | 87·3 | 84·6 | 51 | 70 |
| Mother liquor separated after cooling .... | 71·00 | 72·50 | 57·5 | 58·4 | 6·50 | 2·40 | 82·3 | 79·2 | 28 | — |
| Masse cuite (pan ¼ full) .. .. .. .. | 90·50 | 91·7 | 77·3 | 79·0 | 5·50 | 2·09 | 87·3 | 84·3 | 57 | 71 |
| ,, (pan ½ full) .. .. .. .... | 90·57 | 91·8 | 78·2 | 79·9 | 5·40 | 2·10 | 88·2 | 85·2 | 61 | 71 |
| ,, (pan ¾ full) .. .. .. .. | 91·37 | 92·57 | 77·4 | 78·9 | 5·40 | 2·15 | 86·3 | 84·7 | 62 | 71 |
| ,, (pan quite full) .. .. .... | 91·37 | 92·6 | 79·4 | 81·0 | 5·40 | 2·12 | 88·6 | 85·7 | 62 | 71 |
| Molasses (separated hot) .. .. .. .. | 78·97 | 82·6 | 52·0 | 55·8 | 12·50 | 5·93 | 70·7 | 63·0 | 62 | — |
| ,, (after cooling and filtration) .... | 78·63 | 82·25 | 51·8 | 55·6 | 12·41 | 5·95 | 70·7 | 62·9 | 28 | — |

**Solubility of the Sucrose before and after Graining.**

The concentrated syrup therefore contained 19·57 per cent. of water and 70·21 per cent. of sucrose at the graining point, or 358·8 parts of sucrose per 100 parts of water. At the temperature of 51° C. sucrose dissolves in water in proportion of 72·44 : 27·56. When this syrup had completely crystallized, showing the normal solubility of sucrose in the water present, the said water would have been able to dissolve $\frac{19{\cdot}57 \times 72{\cdot}44}{27{\cdot}56} = 51{\cdot}44$ per cent. of sucrose at 51°C., and $70{\cdot}21 - 51{\cdot}44 =$ 18·77 per cent. would crystallize out. In reality the figure for the solubility of sucrose is much less than the theoretical solubility, as is seen from the analysis of the mother liquor, which was obtained later at a temperature of 28°C. In this liquor, 29 parts of water contained 58·4 parts of sucrose, or 201·4 parts of sucrose per 100 parts of water. At that temperature, 100 parts of pure water can hold 215·3 parts of sucrose in solution. After complete cooling, only 39·41 per cent. sucrose is retained in solution, and 30·80 per cent. has crystallized out.

The syrup used in this experiment being of constant composition, the analyses of the contents of the pan do not show much variation at different periods. The molasses separated hot from the last sample contained 55·8 parts of sucrose on 21·03 parts of water, or 265·3 parts of sucrose per 100 parts of water at 62°C. The solubility of sucrose in pure water at that temperature is as 25·42 : 74·58 or as 100 : 293·4. Again the solubility of sucrose is less in the impure water of the molasses than in pure water. The cooled molasses was rendered turbid by minute sugar crystals, which separated during cooling, and which were so minute that they could not be separated by filtration through asbestos or glass-wool, but passed into the filtrate. Consequently, the analyses of the filtered molasses gave almost the same results as the molasses before filtration. In this impure liquid the influence of temperature on the solubility of sucrose was the same as in pure water. On 100 parts of water 293·4—215·3 = 78·1 parts of sucrose

must have crystallized out, or $\frac{78{\cdot}1 \times 21{\cdot}03}{100} = 16{\cdot}43$ on 100 parts of molasses. The extremely small size of the crystals prevented their direct estimation, so that it is impossible to say whether this figure was actually obtained, but the quantity did not appear to exceed a few units per cent. Hence, in these solutions the ratio of sucrose to water is not only influenced by the normal solubility of pure sucrose in pure water, but also by other circumstances of a much more complicated nature.

II.—Masse Cuite yielding White Sugar.

| Nature of the Sample and Stage of the Boiling Process. | Dry Substance. | Brix. | Polarization. | Sucrose (Clerget). | Reducing Sugar. | Ash. | Actual Purity. | Apparent Purity. |
|---|---|---|---|---|---|---|---|---|
| Concentrated syrup at the graining point | 80·5 | 81·5 | 72·1 | 72·18 | 4·93 | 1·50 | 89·66 | 88·34 |
| The cold molasses after crystallization. | 70·68 | 72·4 | 60·87 | 61·10 | 5·74 | 1·84 | 86·45 | 84·09 |
| Masse cuite, pan ¼ full | 89·36 | 90·49 | 80·00 | 80·16 | 5·43 | 1·63 | 89·70 | 88·40 |
| Molasses contained therein | 77·62 | 80·07 | 59·85 | 60·42 | 8·15 | 3·16 | 77·84 | 75·0 |
| Masse cuite, pan ½ full | 89·18 | 90·13 | 79·92 | 80·10 | 5·34 | 1·65 | 89·98 | 88·67 |
| Molasses contained therein | 75·41 | 77·63 | 57·65 | 58·99 | 8·40 | 3·15 | 78·23 | 75·55 |
| Masse cuite, pan ¾ full | 90·21 | 90·82 | 82·42 | 82·60 | 5·01 | 1·44 | 91·56 | 90·94 |
| Molasses contained therein | 78·21 | 90·55 | 61·54 | 63·03 | 9·00 | 3·25 | 80·59 | 76·40 |
| Masse cuite (pan full) | 88·63 | 89·91 | 80·56 | 80·75 | 5·35 | 1·48 | 90·50 | 89·39 |
| Molasses separated hot | 78·76 | 80·74 | 58·96 | 59·18 | 10·63 | 3·56 | 75·14 | 73·02 |

**Amount of Sucrose Crystallizing in the Pan.**

It is a fact that the quantity of sugar crystallizing from a masse cuite increases as the water-content diminishes, although this increase is not proportional to the increase in concentration. The more a masse cuite is concentrated, the greater the quantity of sugar that crystallizes, but in practical working certain circumstances prevent the concentration proceeding beyond a certain limit, viz., about 6 to 8 per cent. of water, causing the composition of a first masse cuite, boiled from average juice, to be:—

| | |
|---|---|
| Sucrose | 81·10 |
| Glucose | 6·93 |
| Ash | 1·12 |
| Water | 8·66 |
| Undetermined | 2·19 |
| | 100·00 |

A masse cuite from impure juice may give the following figures:—

| | |
|---|---|
| Sucrose | 74·10 |
| Glucose | 11·07 |
| Ash | 1·54 |
| Water | 9·02 |
| Undetermined | 4·27 |
| | 100·00 |

But, though it may appear absurd, the quantity of crystallized sugar does not vary very much in the two cases, provided that the amount of water is approximately the same, as sugar is much more soluble in the water contained in a pure masse cuite than in that of an impure one.

We may assume that in a pure masse cuite, after being completely cooled, one part of water holds two parts of sucrose in solution, so that in the masse cuite containing 8·66 parts of water 17·32 parts of sucrose are in a state of solution and 81·20 − 17·32 = 63·78 as crystals.

**Limits of the Concentration.**

If we could concentrate this masse cuite further, every part of water which is evaporated should cause two parts of sucrose to crystallize, so that in a masse cuite containing 3 per cent. of water, only 6 per cent. of sugar would remain dissolved and the balance be present in the crystallized form. It is, however, impossible to reach such high concentrations unless some radical changes are made in the *modus operandi*, because the last portions of sucrose to crystallize would not be deposited on the crystals already formed, but between them, as "false grain," owing to the total cessation of circulation in so stiff a mass. We have already remarked that perfect circulation is an essential condition of crystalline growth. Moreover, in the absence of circulation, those portions of the masse cuite which are in actual contact with the steam-coils, or other heating surfaces, would become over-heated and caramelized, and heat would cease to be transmitted to the more distant portions of the masse cuite. Finally, it is not possible to get such a stiff masse cuite out of the pan, and for these several reasons one is compelled to leave so much water in the masse cuites, that 12-18 per cent. of the sugar remains dissolved and is removed in the molasses during curing. But, as an appreciable portion of that sugar could be recovered as first sugar (owing to the fact that it passes over into the molasses as syrup), many devices have been suggested for increasing the fluidity of masse cuites and the circulation in the pan so as to induce a part of the dissolved sugar to deposit on the crystals already formed, and thereby be recovered in the first sugars.

It is, of course, quite as impossible to recover all of this dissolved sugar, as it is to clarify juices so thoroughly that a pure sugar solution results. The juices will

always contain certain impurities which have to be separated from the sugar crystals and this separation is brought about by converting the sucrose into a solid (crystal) whilst the impurities remain in solution, so that a syrupy liquid always remains in contact with the crystals. The chief constituent of the impurities, viz., salts, combine with sucrose to form uncrystallizable compounds, which are not dissociated at high concentrations; so that the sucrose so combined is always uncrystallizable and therefore lost in the molasses. Further, it is possible that the molasses may contain other water than that existing in chemical combinations, and such excess water will also hold sucrose in solution and cause a loss on curing.

**Sucrose is present both in a Combined and Free State.**

Whereas that portion of sucrose, which is combined with salts, is unavailable, the second portion may be recovered as first product after evaporating all of the free water (*i.e.*, the water that is not chemically combined in the combinations of sucrose and salts). We have already remarked that too high a concentration of the masse cuite hinders circulation and the regular growth of the sugar crystals, so that mere evaporation of water is not sufficient. We can, however, increase the fluidity of the highly concentrated masse cuite by diluting same with molasses from a previous operation, thereby separating the crystals from one another and enabling them to move freely and to grow regularly.

**Promotion of Circulation by the Addition of Molasses.**

The free, and also part of the combined water are driven off, causing the sucrose to deposit on the existing crystals and being thereby obtained in the solid state. At the end of the concentration the whole of the free sucrose will have crystallized, and the mother-liquor (molasses) will consist of a syrupy liquid containing compounds of organic and inorganic salts with sucrose, glucose, and fructose. In most cases these compounds have lost part of their combined water, which has been driven off owing to the high temperatures, and on the other hand they keep in dissolution some free sucrose, free reducing sugar, gummy matter, gypsum, and other impurities.

**Division of the Masse Cuite into a Liquid and a Solid Portion.**

A part of the dissolved free sucrose crystallizes out on cooling, when there is sufficient crystalline surface upon which it may deposit, and such crystallization is more complete in the case of fine than of coarse grained masse cuite. If this cooling is conducted slowly and regularly one may succeed in obtaining all the free sucrose in the crystallized form, while the saline compounds of sucrose and reducing sugars remain dissolved in the syrupy mother-liquor. The large volume of this latter (sometimes amounting to 60 per cent. of the weight of the masse cuite) keeps the masse cuite fluid even when part of the combined water is driven off, so that the sugar crystals move freely in the mother-liquor and grow regularly.

**Practical Disadvantages of Complete Crystallization of First Masse Cuite.**

But, in order to attain this end, a very copious addition of molasses to the syrup masse cuite is necessary and the capacity of the vacuum pan is diminished proportionally by the presence of this uncrystallizable material. Moreover, as much time and storage capacity are required to allow the last portions of dissolved sucrose to crystallize out from the hot mother-liquor, this method of effecting complete crystallization of first masse cuites has had to be abandoned and other methods substituted for it.

**Various Methods of adding Molasses.**

In the first attempts, the concentrated syrup masse cuite was mixed with about half its volume of hot molasses of about 80° Brix, and the mixture boiled for a quarter of an hour, and subsequently cooled in motion.

As the molasses obtained on curing were of the same composition and purity as the molasses which were added, this last had not lost any sugar, and only served for dilution. The advantages ascribed to this system are that a diluted masse cuite can be discharged from the pan much more rapidly than a stiff one, and that steaming out is obviated by the absence of hard crusts on the coils, and adhering to the walls of the pan. A better yield of sugar is obtained on curing, and of better quality than from undiluted masse cuite. But, as the purity of the molasses added to the masse cuite is the same as that yielded by it (viz., from 55 to 60), the increased yield cannot be as great as the inventors claimed.

**Molasses only used for Dilution.**

A great improvement was effected by concentrating the diluted masse cuite to 93-94 Brix, which yielded molasses of 50 purity. Some of the dissolved sucrose in the added molasses then crystallized out, thereby increasing the yield of the total masse cuite. This improved yield necessitated a decreased yield of second sugar, but as first sugar fetched a higher price, the advantage was perceptible. In any case, there still remained a second product, because the volume of diluting molasses and the required storage capacity would become too great if the dilution were pushed so far that the finished masse cuite yielded first sugar and exhausted molasses. For if the quotient of purity of the finished masse cuite had to be reduced as low as 60 by diluting masse cuite of 86 purity with molasses of 45 purity, only one-third of the pan capacity would be available for boiling syrup (undiluted).

**Concentration of the mixed Masse Cuite.**

In order to avoid these inconveniences, and yet obtain only first sugar and exhausted molasses, crystallization is effected in two stages. The syrup masse cuite is diluted in the pan with molasses of 45 purity, so that the purity of the mixture amounts to 75, after which it is concentrated to 93° Brix. It is cured hot (before being completely crystallized) and the great bulk of the sugar thus recovered as first product without loss of time or resorting to coolers. The molasses obtained on curing

**Complete Crystallization in Two Stages.**

has also a purity of 45, so that the added molasses only served for dilution without being deprived of any sugar. In addition to the molasses added to the masse cuite, and which is recovered unchanged in composition and purity, we also obtain molasses of similar composition originating from the syrup in the masse cuite, so that after the separation of the sugar, the total amount of molasses of 45° purity is increased. This is re-boiled and crystallized, so as to yield exhausted molasses of 30 degrees purity once or twice a day, depending on the quantity of molasses and the capacity of the vacuum pans. To this end, a small quantity of syrup is boiled to masse cuite, and mixed with as much molasses of 45 purity as will reduce the purity of the mixture to 60. The mixture is concentrated to 95° Brix, or even higher, discharged into coolers, and cooled slowly in motion down to 45° C. After two or three days' cooling in motion, this masse cuite is cured and then yields sugar of similar quality and appearance as the first product, and exhausted molasses of 30 quotient, which passes out of the manufacture. When the work is well-conducted, as much exhausted molasses of 30 purity is removed every day as daily enters into the factory in the juice in the form of impurities or non-saccharine matter, and the quantity of molasses of 45° purity which circulates in the factory is just sufficient for the necessary dilutions and admixtures.

**Decomposition of Molasses by repeated Concentrations.**

Consequently, in this process, a part of the molasses never leaves the factory but is repeatedly returned to the pans, and being partially decomposed by repeated heating and concentration, it becomes acid and gummy, and contains so many impurities that it is no longer possible to reduce the purity of the exhausted molasses to the desired point, as at the commencement of the operations. Moreover, the cured sugar develops an unpleasant smell owing to the adhering sour molasses and might deteriorate during storage or transport. When such molasses was boiled separately, and the process started anew with fresh molasses, the purity of the exhausted molasses at once dropped to the desired figure of 30, and this induced manufacturers to avoid keeping molasses in circulation for too long a time.

**Mode of Avoiding Decomposition of Molasses.**

This is accomplished in the following manner: a masse cuite boiled from syrup is cooled and cured, yielding 30 per cent. of its weight of molasses of, say, 70 purity. This molasses is added to a second syrup masse cuite, the mixture concentrated, cooled, and cured, yielding molasses of about 60 purity. This is again mixed with a syrup masse cuite until the resulting molasses reaches the purity of exhausted molasses (about 30). Now, all the molasses in circulation is added to syrup masse cuite so that the mixture has a purity of 60. The molasses obtained on curing this are exhausted, and can be removed and the process started anew with fresh syrup. In this way, all the available sugar may be obtained as first product after 4 or 5 days' work, leaving an exhausted molasses, the quantity of

molasses remaining in circulation being small, so that overheating, sourness, and accumulation of decomposition products are avoided.

**"Absolute Recovery" Process.**

These operations have been further modified and led to the so-called "absolute recovery" process, now to be described in detail.

In order to reduce the purity of the first molasses, that of the syrup may be reduced to 80 by mixing it with first molasses of, say, 60 purity. The resulting masse cuites of 80 purity will yield molasses of about 60 purity. When the syrup is not higher than 80, this mixing with molasses is omitted, but in the case of adding the concentrated molasses to the syrup care must be taken not to concentrate the latter to the same degree as when no molasses are mixed with it, for the concentrated molasses would raise the density of the mixture so high that too small a grain would result. The masse cuite, having a purity of 60, is cured hot, and yields first sugar and molasses of the same purity as that used for mixing with syrup, viz., 58-60. Sufficient of this molasses is drawn into a second masse cuite (of mixed syrup and first molasses of 60 purity) that the purity of the mixture becomes 70; this is also concentrated and cured without cooling, and yields first sugar and a second molasses of 48-50 purity. Finally this is added to a third masse cuite of 80, until the mixture has a purity of 60. This is highly concentrated, cooled gradually, and, on curing, yields sugar and exhausted molasses.

**Particulars of the Process.** A few particulars of this process may be mentioned here.

Before returning the molasses to the pan, they are heated with steam to a higher temperature than that of the already formed masse cuite in the pan, because, otherwise, they would not mix properly with the masse cuite, and by suddenly cooling it, a part of the dissolved sucrose would be precipitated as a "false grain." The molasses are steamed before being drawn into the pan, and the froth removed by means of wooden ladles. Steaming renders the molasses more dilute and easier to handle, it dissolves the fine grain usually present in molasses, and thereby eliminates the possible formation of a secondary crystallization, which causes much trouble in the centrifugals by choking the liners.

In some factories the molasses are diluted with water but in most cases the steaming causes sufficient dilution. The steaming pipe must be perforated on its under surface, otherwise it may get choked by subsiding impurities when the steaming is temporarily stopped. As the heavy impurities remain in the molasses, such steaming and skimming operations are not very effective, and it would be far better if the molasses could be completely clarified and thus rid of all suspended impurities. As cane molasses are much too gummy for subsidation or filtration, the author suggests experiments with a high-speed centrifugal separator (2000 revolutions per minute) by means of which the insoluble impurities might be deposited against the interior of the drum and the clear molasses passed out.

**Boiling of Masse Cuites to yield "Green" Molasses.**

To obtain "green" molasses direct from first masse cuites, or from a mixture of syrup and molasses masse cuites, hot molasses are drawn in slowly and the mixture concentrated, these operations being repeated until the pan is full. For masse cuites of 60 purity the final concentration is 91° Brix, and for those of 70 purity about 93° Brix. The addition of molasses does not require as much supervision as that of syrup, because if once the syrup masse cuite is well boiled and free from false grain, the latter is not so easily formed during the addition and the concentration of the molasses.

**Calculation of the Proportion of Masse Cuite from Syrup and from Molasses.**

The quantity of syrup masse cuite which should be in the pan before molasses are drawn in, that is to say, the ratio between the masse cuite boiled from syrup and from "green" molasses, will depend on the purities of these two constituents and on the use made of the resulting molasses.

Pasma* gives the following relations between the purity of mixed masse cuites and the molasses obtained on curing without previous cooling:—

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| Masse cuite of | 90 | purity | yields a | molasses of | 75 | quotient. | |
| ,, | 88 | ,, | ,, | ,, | ,, | 71 | ,, |
| ,, | 85 | ,, | ,, | ,, | ,, | 65 | ,, |
| ,, | 82 | ,, | ,, | ,, | ,, | 62 | ,, |
| ,, | 80 | ,, | ,, | ,, | ,, | 60 | ,, |
| ,, | 70 | ,, | ,, | ,, | ,, | 50 | ,, |
| ,, | 65 | ,, | ,, | ,, | ,, | 45 | ,, |

When it is necessary to add a molasses of 50 purity to a subsequent masse cuite, the masse cuite yielding such molasses must have a purity of 70, and so on. But, if it is customary to use molasses of a fixed purity for all kinds of masse cuite, the purity of the juice or syrup will decide the quantity of syrup masse cuite which must be present in the pan when the molasses are added.

For example, supposing that molasses of 50 purity are usually added to the second masse cuite, the mixed masse cuite should then have a purity of 70, and we get this by mixing $x$ parts of syrup masse cuite of purity $a$ with $100-x$ parts of molasses masse cuite of 50 purity. We see that the value for $x$ depends on that for $a$ (purity of the syrup) and that the percentage of syrup masse cuite that must be present in the pan before the molasses are drawn in, to form a mixed masse cuite of 70 quotient, may be calculated from the formula:—

$$a \times x + 50\,(100-x) = 100 \times 70$$
$$a \times x - 50\,x = 7000-5000$$
$$x = \frac{2000}{a-50.}$$

This percentage becomes $\frac{2000}{85\text{-}50} = 57{\cdot}2$ for a purity of the syrup $= 65$, and $\frac{2000}{80\text{-}50} = 66{\cdot}7$ for one of 80.

* *Archief voor de Java Suikerindustrie*, 1904, 624.

This means that with syrup of 65 purity, 57·2 per cent. of the mixed contents of the pan must consist of syrup, and the balance of hot molasses. With syrup of 60 purity, this percentage becomes 66·7, and so forth.

Where the same pans are always used, it is advisable to calculate once for all the height to which each must be filled with syrup masse cuite for different purities of syrup and of mixed masse cuite, and to mark the outside of the pan, so that the pan boiler may fill the pan with syrup masse cuite to a certain mark in every instance before drawing in molasses.

**Masse Cuites yielding "Green" Molasses are cured hot.**

When molasses have to be re-boiled in the course of manufacture, the masse cuites yielding such molasses need not be completely crystallized, and are therefore usually cured hot to save time and storage space. The purity of the molasses so obtained without previous cooling is known by experience, and serves as a basis for calculating the proportions between the two components.

This, however, is not the case with masse cuites yielding exhausted molasses, where every effort should be made to induce as much sucrose to crystallize as possible, and in the most favourable form to be recovered, because any sugar which fails to crystallize or which crystallizes as minute crystals which cannot be separated in the centrifugals, disappears in the exhausted molasses and is lost. The boiling, cooling, and curing of the molasses masse cuites, therefore, require much attention and skill, also suitable plant, since any losses which occur cannot be rectified.

**Masse Cuites yielding exhausted Molasses are cooled.**

The vacuum pan used for boiling final masse cuites should not be deep, the proof-stick should be fixed as low as possible, the sight-glasses extended to the bottom, and a good circulation maintained. These conditions are necessary to prevent the concentrated masse cuite settling to the bottom, instead of mixing with the molasses, and thus frustrating the object aimed at. Pans provided with steam chambers, so called calendrial pans, are not suitable for boiling these masse cuites, owing to the difficulty of gauging the quantity of syrup masse cuite in the pan before introducing the molasses.

**Shape of the Pan for boiling Molasses.**

When boiling a molasses masse cuite, we first calculate the proportion of syrup masse cuite and molasses to be added to yield a mixture of 60 purity. Usually the proportion of syrup masse cuite is from one-quarter to one-third of the total volume. Such a masse cuite must be kept free from false grain, and care must be taken to form sufficient grain as will ensure a proper proportion of fully developed crystals in the finished masse cuite. The crystals increase in size during boiling but their number should not increase. It is therefore advisable to grain high up, so that after two or three additions of syrup and concentration, sufficient masse cuite is in hand, and the introduction of molasses commenced. The molasses are drawn

**Method of Boiling Final Masse Cuites.**

in slowly and regularly, in not too large quantities at a time. The mixed masse cuite is boiled at a rather high temperature, viz., 66° C. (150·8° F.) at a vacuum of 70 c.m. (27·5 inches) and concentrated to about 96° Brix. Just before discharging, the masse cuite is warmed to 70° C. (158° F.) in order to facilitate its discharge from the pan, by slackening the speed of the air-pump and diminishing the injection. The steam supply being then turned off, and the air-pump and injector stopped, air is admitted to the pan and the hot masse cuite discharged into coolers by opening the bottom door of the pan.

**Composition of Masse Cuites and the Molasses obtained therefrom.**

A number of analyses of masse cuites and the contained molasses at various stages of the boiling process are recorded here. They illustrate the composition of the contents of the pan and the gradual exhaustion of the mother-liquor during boiling.

I.—Masse Cuites Mixed with Molasses and Yielding "Green" Molasses.

| No. | Nature of the Sample and Stage of the Process. | Brix. | Dry Substance. | Polarization. | Sucrose (Clerget). | Reducing Sugar. | Ash. | Apparent Purity. | Real Purity. |
|---|---|---|---|---|---|---|---|---|---|
| 1 | Syrup .. .. .. .. .. .. .. .. | 59·96 | 59·87 | 54·4 | 54·61 | 2·92 | 0·82 | 90·72 | 91·42 |
| 2 | The same at the graining point .. .... | 82·06 | 81·35 | 75·42 | 75·23 | 3·65 | 1·00 | 91·90 | 92·41 |
| 3 | The same more concentrated .. .. .. | 88·28 | 88·09 | 80·96 | 80·96 | 3·97 | 1·16 | 91·70 | 91·90 |
| 4 | At the point where molasses should be added | 90·67 | 90·03 | 83·76 | 83·90 | 4·85 | 1·32 | 92·37 | 93·19 |
| 5 | Mother-liquor.. .. .. .. .. .. .. | 79·47 | 77·24 | 60·22 | 61·01 | 8·57 | 3·09 | 75·47 | 79·13 |
| 6 | Molasses added .. .. .. .. .. .... | 75·82 | 72·88 | 56·16 | 57·06 | 9·86 | 2·66 | 74·07 | 78·29 |
| 7 | Mixture after two additions of molasses.. | 91·0 | 89·22 | 79·8 | 79·2 | 5·50 | 1·61 | 87·71 | 88·77 |
| 8 | Mother-liquor .. .. .. .. .. .... | 77·09 | 73·56 | 54·51 | 55·62 | 8·30 | 3·33 | 70·71 | 75 61 |
| 9 | Molasses added .. .. .. .. .. .. | 75·74 | 70·76 | 55·22 | 56·12 | 9·34 | 2·85 | 73·17 | 79·31 |
| 10 | Mixture after three additions of molasses. | 91·88 | 89·23 | 77·40 | 77·72 | 8·84 | 1·96 | 84·23 | 87·20 |
| 11 | Mother-liquor.. .. .. .. .. .. .. | 84·2 | 77·49 | 49·99 | 51·39 | 10·81 | 4·11 | 59·37 | 66·31 |
| 12 | Masse cuite finished.. .. .. .. .... | 91·22 | 72·88 | 75·83 | 57·06 | 9·74 | 2·01 | 83·13 | 84·46 |
| 13 | Molasses contained in it .. .. .. .. | 82·13 | 77·37 | 50·19 | 51·65 | 10·92 | 4·23 | 60·94 | 66·76 |
| | II. | | | | | | | | |
| 1 | Concentrated syrup at the graining point. | 81·7 | 80·01 | 70·34 | 71·12 | 4·70 | 1·91 | 86·1 | 88·9 |
| 2 | The same concentrated .. .. .. .... | 90·3 | 88·9 | 77·6 | 78·7 | 5·2 | 2·12 | 85·9 | 88·6 |
| 3 | Mother-liquor.. .. .. .. .. .. .. | 78·7 | 75·2 | 48·6 | 49·1 | 11·4 | 4·80 | 61·7 | 65·3 |
| 4 | Molasses used for mixing .. .. .... | 84·2 | 80·39 | 52·0 | 52·7 | 11·0 | 4·90 | 61·7 | 65·1 |
| 5 | Mixture after one addition.. .. .. .. | 91·3 | 89·45 | 74·2 | 76·4 | 6·76 | 2·90 | 81·3 | 85·4 |
| 6 | Mother-liquor .. .. .. .. .. .... | 82·0 | 78·5 | 48·0 | 51·1 | 14·5 | 5·19 | 58·5 | 65·0 |
| 7 | Masse cuite finished .. .. .. .. .. | 91·4 | 89·54 | 64·0 | 67·3 | 6·4 | 2·81 | 70·0 | 75·2 |
| 8 | Molasses contained in it.. .. .. .... | 82·1 | 78·49 | 39·0 | 43·9 | 14·3 | 5·18 | 47·5 | 55·9 |

We here notice a constant lowering of the quotient of purity of the mother-liquor in proportion as the evaporation of water proceeds, such evaporation being favoured by the better circulation after each addition of molasses.

The molasses separated from the above masse cuites are mixed with syrup masse cuites in other pans to form the following mixtures:—

MASSE CUITES YIELDING EXHAUSTED MOLASSES.

| Nature of the Sample and Stage of the Procees. | Dry Subtance. | Brix. | Polarization. | Sucrose (Clerget). | Reducing Sugar. | Ash. | Real Purity. | Conventional Purity. | Temperature. | Vacuum. |
|---|---|---|---|---|---|---|---|---|---|---|
| I. | | | | | | | | | | |
| Concentrated primary masse cuite | 87·9 | 89·6 | 73·6 | 75·9 | 5·75 | 2·52 | 89·8 | 82·2 | — | — |
| Molasses used for mixing.. .... | 79·3 | 84·1 | 38·2 | 43·3 | 21·3 | 7·30 | 54·6 | 45·4 | — | — |
| Mixed masse cuite after 1 addition | 91·0 | 93·6 | 64·8 | 69·6 | 12·5 | 4·28 | 76·5 | 69·2 | 48 | 71 |
| Mother-liquor .. .. .. .... | 81·4 | 88·9 | 36·6 | 42·3 | 21·3 | 7·12 | 51·9 | 42·6 | 48 | — |
| Mixed masse cuite after 2 additions | 92·0 | 95·0 | 61·4 | 66·7 | 14·7 | 4·91 | 72·5 | 64·6 | 56 | 71 |
| Mother-liquor .. .. .. .... | 83·6 | 88·5 | 33·2 | 40·3 | 23·8 | 7·93 | 48·2 | 37·5 | 56 | — |
| Mixed masse cuite after 4 additions | 91·9 | 94·7 | 62·0 | 66·8 | 13·5 | 4·37 | 72·7 | 65·4 | 60 | 71 |
| Mother-liquor .. .. .. .... | 84·8 | 89·9 | 31·8 | 39·5 | 25·6 | 8·28 | 46·6 | 35·3 | 60 | — |
| Mixed masse cuite finished .. .. | 92·7 | 95·4 | 67·2 | 70·2 | 12·5 | 4·39 | 75·7 | 70·4 | 66 | 71 |
| Mother-liquor .. .. .. .... | 82·4 | 87·6 | 33·6 | 40·3 | 25·6 | 8·23 | 48·9 | 38·4 | 66 | — |
| Molasses separated after cooling .. .. .. .. .. .. | 82·0 | 88·3 | 25·0 | 31·8 | 30·1 | 10·2 | 38·8 | 28·3 | 40 | — |
| II. | | | | | | | | | | |
| Concentrated primary masse cuite | 89·6 | 91·2 | 75·4 | 77·9 | 6·— | 2·50 | 87·0 | 82·7 | — | — |
| Molasses used for mixing.. .... | 77·8 | 81·6 | 42·2 | 44·8 | 16·1 | 6·23 | 57·6 | 51·7 | — | — |
| Mixed masse cuite after 1 addition | 92·5 | 94·9 | 72·2 | 73·7 | 9·6 | 3·65 | 79·7 | 76·1 | 54 | 73 |
| Mother-liquor .. .. .. .... | 81·6 | 86·5 | 39·6 | 44·0 | 20·9 | 7·95 | 53·9 | 45·8 | 54 | — |
| Mixed masse cuite after 3 additions | 92·8 | 95·3 | 67·4 | 69·6 | 10·9 | 4·21 | 75·2 | 70·7 | 58 | 71 |
| Mother-liquor .. .. .. .... | 84·2 | 89·6 | 36·2 | 42·0 | 22·8 | 8·69 | 49·9 | 40·4 | 58 | — |
| Mixed masse cuite after 5 additions | 92·6 | 95·3 | 63·0 | 65·5 | 12·5 | 4·77 | 70·7 | 66·1 | 66 | 71 |
| Mother-liquor .. .. .. .... | 86·3 | 91·7 | 33·4 | 39·8 | 23·3 | 8·87 | 46·1 | 36·4 | 66 | — |
| Mixed masse cuite finished .. .. | 94·1 | 97·4 | 57·6 | 61·6 | 13·9 | 5·28 | 65·5 | 59·2 | 68 | 71 |
| Mother-liquor .. .. .. .... | 84·7 | 90·7 | 30·6 | 37·3 | 24·4 | 9·55 | 44·0 | 33·7 | 68 | — |
| Molasses separated after cooling .. .. .. .. .. .. | 83·2 | 89·5 | 24·0 | 31·6 | 25·0 | 10·05 | 38·0 | 27·0 | 40 | — |
| III. | | | | | | | | | | |
| Concentrated primary masse cuite | 88·3 | 90·0 | 72·2 | 74·8 | 8·2 | 2·79 | 84·7 | 80·2 | — | — |
| Molasses used for mixing.. .... | 74·0 | 78·8 | 37·6 | 42·4 | 16·9 | 7·65 | 57·3 | 47·8 | — | — |
| Mixed masse cuite after 1 addition | 92·4 | 94·8 | 68·8 | 72·2 | 7·46 | 3·92 | 78·2 | 72·6 | 52 | 71 |
| Mother-liquor .. .. .. .... | 83·8 | 88·6 | 40·3 | 45·2 | 14·4 | 7·67 | 53·9 | 45·5 | 52 | — |
| Mixed masse cuite after 3 additions | 92·5 | 95·5 | 61·8 | 66·2 | 9·1 | 4·81 | 71·6 | 64·7 | 56 | 71 |
| Mother-liquor .. .. .. .... | 82·2 | 87·8 | 36·2 | 42·6 | 20·0 | 8·49 | 51·8 | 41·2 | 56 | — |
| Mixed masse cuite after 5 additions | 92·2 | 95·7 | 61·1 | 65·4 | 11·1 | 5·39 | 70·9 | 63·7 | 59 | 71 |
| Mother-liquor .. .. .. .... | 86·0 | 92·0 | 33·0 | 40·1 | 22·5 | 9·51 | 46·6 | 35·9 | 59 | — |
| Mixed masse cuite finished .. .. | 91·4 | 95·9 | 56·3 | 61·4 | 15·6 | 7·02 | 67·2 | 58·7 | 66 | 71 |
| Mother-liquor .. .. .. .... | 85·3 | 91·4 | 30·6 | 37·8 | 22·4 | 9·43 | 44·5 | 33·4 | 66 | — |
| Molasses separated after cooling .. .. .. .. .. .. | 80·04 | 86·2 | 23·8 | 32·0 | 23·8 | 9·65 | 40·0 | 27·5 | 40 | — |

MASSE CUITES YIELDING EXHAUSTED MOLASSES.—*Continued.*

| Nature of the Sample and Stage of the Process. | Dry Substance. | Brix. | Polarization. | Sucrose (Clerget). | Reducing Sugar. | Ash. | Real Purity. | Conventional Purity. | Temperature. | Vacuum. |
|---|---|---|---|---|---|---|---|---|---|---|
| IV. | | | | | | | | | | |
| Concentrated primary masse cuite | 88·6 | 90·2 | 74·2 | 76·6 | 5·88 | 2·61 | 84·7 | 80·2 | — | — |
| Molasses used for mixing.. .... | 77·6 | 81·5 | 38·1 | 44·0 | 15·4 | 6·40 | 56·7 | 49·1 | — | — |
| Mixed masse cuite after 1 addition | 91·4 | 93·8 | 65·6 | 69·8 | 9·4 | 3·87 | 76·5 | 69·9 | 58 | 70 |
| Mother-liquor .. .. .. .... | 81·6 | 87·44 | 37·2 | 43·6 | 17·8 | 7·20 | 53·4 | 42·5 | 58 | — |
| Mixed masse cuite after 2 additions | 89·95 | 92·8 | 59·4 | 64·1 | 11·6 | 4·71 | 71·2 | 64·0 | 60 | 71 |
| Mother-liquor .. .. .. .... | 80·10 | 83·8 | 33·3 | 40·7 | 14·8 | 5·98 | 50·3 | 39·7 | 60 | — |
| Mixed masse cuite after 4 additions | 89·3 | 92·7 | 56·8 | 61·4 | 12·8 | 5·10 | 68·7 | 61·2 | 66 | 71 |
| Mother-liquor .. .. .. .... | 84·4 | 89·2 | 33·2 | 40·5 | 20·0 | 7·98 | 48·0 | 37·2 | 66 | — |
| Mixed masse cuite finished .. .. | 90·9 | 94·5 | 54·4 | 59·4 | 14·3 | 5·81 | 65·3 | 57·9 | 70 | 71 |
| Mother-liquor .. .. .. .... | 84·0 | 90·1 | 29·6 | 37·7 | 22·5 | 9·6 | 46·1 | 32·8 | 70 | — |
| Molasses separated after cooling .. .. .. .. .. .. | 83·2 | 89·4 | 24·6 | 31·6 | 24·1 | 9·9 | 40·4 | 27·5 | 40 | — |

The masse cuites were discharged hot, and this explains why in some cases the purity of the mother-liquor of the finished masse cuites is higher than at a previous stage of the boiling. We again notice a constant lowering of the purity of the mother-liquor as crystallization proceeds. The advantages of cooling are shown by the purity of the molasses obtained after cooling, being about ten points lower than that of the mother-liquor present in the hot masse cuites.

**Crystallization of Sucrose during cooling.**

This decrease in purity is really due to crystallization of sucrose and not to sourness or inversion during cooling, as is proved by the relation between the figures for reducing sugar and ash in the last mother liquor and in the exhausted molasses. If the molasses contained more reducing sugar for the same amount of ash than the final mother-liquor, this would prove that the former had increased at the expense of the sucrose content and quotient of purity. In the examples cited above, these ratios are as under:—

| | I. | II. | III. | IV. |
|---|---|---|---|---|
| Final mother liquor .. .. | 3·01 .. | 2·56 .. | 2·28 .. | 2·34 |
| Exhausted molasses .. .... | 2·95 .. | 2·49 .. | 2·46 .. | 2·43 |

and we are justified in concluding that as the ratios are not perceptibly disturbed, no appreciable inversion or decomposition has taken place during the cooling process, and that the decrease in quotient of purity is not due to decomposition or inversion but to crystallization of sugar.

The four analyses mentioned above are examples of practical working, and show us how the mother-liquors become gradually exhausted with the formation of completely exhausted molasses.

The hot mother-liquors deposited sugar in the sample bottles, but the crystals were too fine to be determined quantitatively.

**Ratio of Sucrose to Water in the Final Molasses.**

The ratio of sucrose to water in the mother liquors and final molasses cannot serve as a measure of the solubility of sucrose in the water present in those liquids, since they chiefly consist of hydrated combinations of sugars and salts, in which the amount of sucrose depends on the amount of reducing sugar simultaneously present and the amount of water on the degree of concentration of the finished masse cuite. It is obvious that if concentration had been carried farther, a few more per cents of water would have been driven off from the saline combination and although no sugar crystallizes out or becomes dissolved, the ratio of sucrose to water would be completely changed.

The above examples from practice show that the concentrations, &c., referred to attain the desired result, since a lower apparent purity of the molasses than 27 will not be possible.

**Calculation of the total Amount of Masse Cuite on 100 parts of Dry Substance from Syrup.**

Pasma* calculated the total quantity of masse cuite obtained on working up the syrup by various methods, for different purities of syrup, viz., 90, 88, 85, and 82, and on a basis of 100 parts of dry substance in syrup and in masse cuites, so that the water present in the different products may be ignored. He classifies the methods of working as follows :—

A. Original method of adding the molasses in two operations (page 227).

*a.* With masse cuites of 65 quotient yielding green molasses and
,, ,, 60 ,, ,, final ,,

*b.* ,, ,, 70 ,, ,, green ,, and
,, ,, 60 ,, ,, final ,,

B. The more modern method of boiling with a minimum quantity of molasses in circulation (page 228).

With masse cuites of 80 and 70 quotient yielding green molasses and
,, ,, 60 ,, ,, final ,,

C. "Absolute recovery" process without circulating molasses (page 229).

With masse cuites of 90, 88, and 70, at a purity of 90
,, ,, 88, 80, ,, 70, ,, 88
,, ,, 85, ,, 70, ,, 85
,, ,, 82, ,, 70, ,, 82
all yielding green molasses and
One masse cuite yielding final molasses and possessing a purity of 60.

All calculations are based on the figures for dry substance as determined by the degrees Brix and on purity as calculated from the degrees Brix and polarization.

* *Archief voor de Java Suikerindustrie*, 1904, 616.

The purity of the raw sugar is taken as a fixed value 97·5 (apparent). In the following formulae we make use of these abbreviations :—

m.c. = dry substance in mixed masse cuite.
s = dry substance in syrup.
ret. m. = dry substance in returned molasses.
cent. m. = dry substance in centrifugalled molasses.
R = dry substance in sugar.

Further, we will suppose that, without cooling, a masse cuite of

| | | | | | | |
|---|---|---|---|---|---|---|
| 90 | purity | yields a | centrifugalled | molasses of | 75 | purity. |
| one of 88 | ,, | ,, | ,, | ,, | 71 | ,, |
| 85 | ,, | ,, | ,, | ,, | 65 | ,, |
| 82 | ,, | ,, | ,, | ,, | 62 | ,, |
| 80 | ,, | ,, | ,, | ,, | 60 | ,, |
| 70 | ,, | ,, | ,, | ,, | 50 | ,, |
| 65 | ,, | ,, | ,, | ,, | 45 | ,, |

The following formulae may be used for calculating the proportion of dry substance from syrup occurring in a mixed masse cuite of a given purity, which is formed by the mixing of that syrup and a molasses of a given purity.

**Fundamental Formulae.**

$$x \times \text{quot. s} + (100 - x) \times \text{quot. ret. m.} = 100 \times \text{quot. m.c.}$$

$$x \text{ or s} = \frac{\text{quot. m.c.} - \text{quot. ret. mol.}}{\text{quot. s} - \text{quot. ret. mol.}} \times 100.$$

The quantity of returned molasses is therefore $100 - x$ or $100 - s$. The amount of sugar which may be obtained from a given masse cuite can be calculated as follows :—

$$x \text{ or R} = \frac{\text{quot. m.c.} - \text{quot. cent. mol.}}{97{\cdot}5 - \text{quot. cent mol.}} \times 100$$

and the amount of molasses obtained on curing becomes $100 - R$.

**Calculation.** The calculation is for a purity of 90 in the syrup for the various methods described above.

A. *a.* 100 m.c. of 65 yield .. .. .. .. .. 61·9 molasses of 45.
For 100 ,, 65 is required .. .. .... 55·6 ,, 45.

100 m.c. of 65 thus yield a surplus of .. 6·3 ,, 45.

100 m.c. of 60 require 66·7 molasses of 45, which are furnished by $\frac{66{\cdot}7 \times 100}{6{\cdot}3} = 1059$ m.c. of 65.

1059 m.c. of 65 are derived from 470·2 m.c.
100 ,, 60 ,, ,, 33·3 ,,

1159 503·5 ,,

In this case 100 parts of syrup masse cuite yield a total of $\frac{1159}{503{\cdot}5} = 230{\cdot}2$ parts of mixed masse cuite.

A. *b*. 100 m.c. of 70 yield .. .. .. .. .. .. 58 mol. of 50.
For 100 ,, 70 are required .. .. .. .... 50 ,, 50.
100 ,, 70 thus yield a surplus of.. .. .. 8 ,, 50.

100 m.c. of 60 require 75 molasses of 50, which are derived from $\frac{75 \times 100}{8} = 938$ m.c. of 70.

938 m.c. of 70 are derived from 469 m.c.
100 ,, 60 ,, ,, 25 ,,
1038 494 ,,

In this case 100 parts of syrup masse cuite yield a total of $\frac{1038}{494} = 210{\cdot}1$ parts of mixed masse cuite.

B. 100 m.c. of 80 yield.. .. .. .. .. .. .. 46·7 mol. of 60.
For 100 ,, 80 are required.. .. .. .. .... 33·3 ,, 60.
100 ,, 80 thus yield a surplus of .. .. .. 13·4 ,, 60.

We saw from A (*b*) that

| | | | | | |
|---|---|---|---|---|---|
| 100 | m.c. of | 70 yield .. .. .. .. | 58 | molasses of | 50 and |
| for 100 | ,, | 70 one requires .. .... | 67 | ,, | 60 ,, |
| 100 | ,, | 60.. .. .. .. .. .. .. | 75 | ,, | 50. |
| 100 | ,, | 60 require .. .. .... | 75 | ,, | 50 and |
| 100 | ,, | 70 yield .. .. .. .. | 58 | ,, | 50; |

so that in order to supply sufficient molasses for 100 m.c. of 60, there are required $\frac{75 \times 100}{58} = 129$ m.c. of 70. For 100 m.c. of 70 are required $\frac{67 \times 100}{13{\cdot}4} = 500$ m.c. of 80, or for 129 m.c. of 70 $\frac{129 \times 500}{100} = 645$ m.c. of 80.

100 m c. of 60 are derived from 25 m.c.
130 ,, 70 ,, ,, 43 ,,
645 ,, 80 ,, ,, 432 ,,

875 mixed m.c. is derived from 500 mixed m.c.;

so that 100 parts of original m.c. from syrup have yielded $\frac{875}{500} \times 100 = 175$ mixed m.c.

C. 100 m.c. of 90 yield .. .. .. .. .. .. 33·3 molasses of 75.
100 ,, 80 ,, .. .. .. .. .... 46·7 ,, 60.
100 ,, 70 ,, .. .. .. .. .. .. 58·0 ,, 50.
For 100 ,, 70 there are required .. ... 66·7 ,, 75.
,, 100 ,, 70 ,, ,, .. .. .. 67·0 ,, 60.
,, 100 ,, 60 ,, ,, .. .... 75·0 ,, 50.

For 100 m.c. of 60 the molasses is furnished by 129 m.c. of 70, for which the molasses is furnished by $\frac{129 \times 67}{46{\cdot}2} = 186$ m.c. of 80. The molasses for this

portion is again furnished by $\frac{186 \times 66{\cdot}7}{33{\cdot}3} = 372$ m.c. of 90.

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| 100 | m.c | of | 60 | are | derived | from | 25 original m.c. |
| 129 | ,, | | 70 | ,, | ,, | | 43 ,, |
| 186 | ,, | | 80 | ,, | ,, | | 62 ,, |
| 372 | ,, | | 90 | ,, | ,, | | 372 ,, |

787 mixed m.c. are derived from 502 original m.c.; or 100 parts of original m.c. have yielded $\frac{787 \times 100}{502} = 157$ parts of mixed masse cuite.

The figures for the purities of syrup of 88, 85 and 82 may be calculated in the same way.

In order to make the calculation complete, the author calculated how much total masse cuite would have come from 100 original m.c. if no molasses were returned, but had been boiled to string proof as second products. In our instance of 90 and 85 purity we reckon 1 m.c. of first sugar for second, 1 for third, and 1 for last sugar, at a purity of 85 and 82; only first, second, and last sugars are made, while at a purity of the syrup of 75, only first and last sugars are made.

| | | | |
|---|---|---|---|
| 100 m.c. of 90 yield | .. .. .. .. | 33·3 molasses of | 75. |
| 100 ,, 75 ,, | .. .. .. .... | 60 ,, | 60. |
| 100 ,, 60 ,, | .. .. .. .. .. | 71 ,, | 45. |
| 100 ,, 45 ,, | .. .. .. .... | 100 last m.c. | |

The total amount is, therefore,

| | |
|---|---|
| 100 first m.c. .. .. .. .. .. .. .. .. .. | = 100 |
| $\frac{100 \times 33{\cdot}3}{100}$ second m.c. .. .. .. .. .. .... | = 33·3 |
| $\frac{33{\cdot}3 \times 60}{100}$ third m.c. .. .. .. .. .. .. .. | = 20·0 |
| $\frac{20 \times 71}{100}$ last m.c. .. .. .. .. .. .. .... | = 14·2 |
| | 167·5 |

total amount of m.c. for 100 original m.c. from syrup.

In the following table the amounts of total dry masse cuite are recorded, calculated on 100 parts of dry substance in syrup for the various quotients of purities and for the various methods of boiling.

| Method. | Quotient of the original Syrup. | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | 90 | | 88 | | 85 | | 82 | | 75 | |
| Without returning Molasses .. .. .. | 167·5 | 100 | 173·— | 103 | 163·5 | 98 | 180·— | 108 | 153·— | 91 |
| A (a).. .. .. .... | 230·— | 138 | 221·— | 132 | 208·— | 124 | 195·— | 116 | 145·— | 87 |
| A (b) .. .. .. .. | 210·— | 125 | 203·— | 121 | 195·— | 116 | 180·— | 108 | 154·— | 92 |
| B .. .. .. ... | 175·— | 105 | 170·— | 102 | — | — | — | — | — | — |
| C .. .. .. .. .. | 167·5 | 100 | 160·— | 95 | 155·— | 93 | 158·— | 95 | 151·— | 90 |

With syrups of high purities the differences in the total quantity of masse cuite for the different methods are considerable, but they decrease with the quotient of purity and become imperceptible at purities of about 70. We see from the table that with usually high purities of cane juice, the methods B and C yield much less masse cuite than either of the methods A, but this advantage diminishes when impurer juices have to be worked up.

The required pan capacity does not bear a direct relation to the quantity of masse cuite expected, because the duration of the boiling process is not identical for each method. It is evident that it takes more time to concentrate a masse cuite consisting only of syrup than one consisting largely of concentrated molasses.

Syrup masse cuites take longer to boil than those to which molasses are added, but actual figures cannot be given because account must also be taken of the time required for filling and emptying the pan. A pan which can be filled and emptied quickly may be charged once more in the same period than another in which those operations require considerable time, so that as every pan has its peculiarities the time required to boil any particular masse cuite cannot be definitely stated. A single example may suffice however.

Supposing that a syrup masse cuite requires 8 hours, then one consisting of half syrup and half molasses will require 7½ hours, a molasses masse cuite, 6, &c., or on the average 7 hours (filling and discharging being included).

To calculate the pan capacity required for a factory crushing 1000 tons of cane daily, we proceed as follows:—

**Calculation of the Pan Capacity.**

1000 tons of cane yield 800 tons of normal juice containing 18 per cent. dry substance, or 144 tons of dry masse cuite, or 158·2 tons of moist masse cuite containing 90 per cent. dry substance. Assuming the purity of the syrup to be 85, and treating this as in scheme C, we have 155 parts of mixed masse cuite for every 100 parts of syrup masse cuite, and for 158·2 tons of the latter, $\frac{158{\cdot}2 \times 155}{100} = 245{\cdot}2$ tons of mixed masse cuite. The specific gravity being 1·513, these 245·2 tons occupy a space of 212 cubic yards, and as the masse cuites generally remain in the pans for 7 hours, the pan capacity for 1000 tons of cane per 24 hours will be $\frac{212 \times 7}{24} = 62$ cubic yards.

**Cases in which Molasses should be returned.**

The advisability of returning molasses to the first masse cuites is determined by the class of sugar to be made. When making white sugar the syrup should be as pure as possible and every contamination with molasses should be strictly avoided. Therefore we are compelled, in this case, to make second sugars too. When making refining crystals, on the contrary, molasses may be safely returned, but we must bear in mind that, in order to ensure a complete exhaustion of the last molasses, the last molasses sugars should be very finely grained.

Impure syrups and molasses cannot be grained, but are boiled smooth or "string proof," in other words, they are so concentrated as to become super-saturated solutions at the temperature prevailing in the vacuum pan. The hot masse cuite is then discharged into tanks or crystallizers, wherein the sugar is allowed to crystallize whilst the masse cuite cools. The boiling is conducted as follows:—A vacuum having been created in the pan, molasses are drawn in until the coils are quite covered. Steam is then admitted, and concentration commenced; molasses being drawn in from time to time, so as to keep the coils constantly covered. Now and again a proof-stick sample is taken from the pan and drawn out between thumb and finger, forming a thread which breaks at a certain length. If the thread breaks too soon, the concentration has not been carried far enough; if it does not break at all, it has gone too far. The author is of opinion that the refractometer will prove a valuable guide as to the exact degree of concentration of the masse cuite. At present, the skill and experience of the pan-boiler alone determines this, partly by the "breaking-length" of the thread, and partly by the appearance of the sample. Generally speaking, first molasses may be more highly concentrated than second or third, while the temperature at which molasses are boiled is rather high.

**Boiling String-proof.**

Whereas, in boiling syrup masse cuites, the object is to induce sugar to crystallize in the pan, and to boil at a relatively low temperature, the object now in view is to concentrate the masse cuite as much as possible without forming crystals in the pan, in order to obtain subsequently the maximum crystallization in the cooling tanks. The higher the temperature of such a molasses masse cuite, the more sugar can it hold in a supersaturated state, and deposit as crystals on cooling. In order to avoid decomposing the reducing sugar, it is advisable not to exceed 70° C. during the boiling. When the masse cuite is sufficiently concentrated, the steam and molasses supply are shut off, the air-pump stopped, and air admitted to the pan. The contents of the pan are then run into tanks or waggons placed under the discharge door.

In the chapter on After Products further particulars will be given concerning the density and other properties of low-grade masse cuites.

Losses of sugar may occur during the boiling process, and may be brought about either by decomposition or by boiling over, both of which causes are more common in the case of molasses masse cuites than with syrup masse cuites.

**Loss of Sucrose during Boiling.**

It is true that in the case of syrup masse cuites the boiling temperature is, as a rule, very low, but local overheating sometimes occurs so that sucrose is decomposed. The author has several times detected notable percentages of caramelized substances in scale from the coils, indicating decomposition of sugar by overheating.

A few analyses calculated to 100 parts of dry substance here follow:—

| Scale on Coils. | Scale from coils in 1st product vacuum pans. | Scale from coils in 2nd product vacuum pans. |
|---|---|---|
| Loss on ignition | 22·70 | 26·40 |
| Silica | 38·12 | 37·46 |
| Phosphoric acid | — | 1·65 |
| Sulphuric acid | 8·20 | 4·74 |
| Carbonic acid | 0·64 | — |
| Iron oxide | 13·29 | 23·34 |
| Alumina | 2·10 | 3·26 |
| Lime | 6·78 | 4·88 |

**Boiling Over.**

The mechanical losses during the boiling of syrup masse cuites are unimportant; the risk of boiling over being small because, when the pan is full, the contents are no longer fluid. This, however, is not the case with molasses masse cuites in which decomposition products sometimes accumulate to such a degree, that spontaneous decomposition may be brought about by local overheating. The temperature then suddenly rises, with the evolution of gases, so that the whole masse begins to foam violently and passes upwards through the "save all" and air-pump into the condensed-water gutter, leaving only a small quantity of masse cuite in the pan.

Although the author is unable to give figures, he is convinced that the mysterious losses of sucrose sometimes experienced when boiling molasses masse cuites are solely due to spontaneous decomposition of primary decomposition products of sugar. This decomposition is brought about by local overheating, and when once started cannot be stopped. As remarked above, the effects are inversion and loss by decomposition, owing to the sudden rise in temperature. This phenomenon will be further discussed in the chapter on fermentation of molasses.

**Concrete Sugar.**

What is known as "concrete sugar" is manufactured in several countries for direct consumption by concentrating syrups until it becomes a hard crystalline mass. The clarified juice is concentrated until a drop of the syrup solidifies when thrown into a pail of water. The mass is then allowed to cool, and become a solid block. It is evident that as the concentration is carried out at a high temperature this must be done under ordinary atmospheric pressure, and great care must be taken to prevent charring.

**Fryer's Concretor.**

In the year 1868 Fryer constructed an apparatus for this purpose, which he called a "concretor." It consisted of a series of shallow trays, set at a slight angle and fitted with transverse partitions, forming baffle-plates, by means of which the juice was caused to flow across each tray six times, and to traverse a total heating surface of 400 feet. Under the highest

tray was a furnace, the heat from which was conducted under the other trays, so that the juice passing through the trays in a constant stream of about ½ inch in depth was caused to boil briskly, and concentrated to a density of 55-60° Brix. After leaving the trays (in from 10-12 minutes) the juice was further concentrated in a rotating drum, fitted internally with curved blades over which the liquor flowed, exposing a large surface to the heated air drawn through it by means of a fan or blower. After about twenty minutes the concentrated liquor was run out of the drum when at a temperature of about 200° F. (93·5° C.), and of such a consistency that it set to a solid mass on cooling. After a short period of success, Fryer's concretor was abandoned on account of the wasteful method of heating by open fire.

**Lillie's Evaporator.**

It was next suggested that the juice might be evaporated in vacuo by multiple-effet, but under ordinary working conditions the temperature of the most concentrated juice was lowest, so that this began to crystallize long before all the water was evaporated. This difficulty was overcome by reversing the action of a triple-effet by introducing the clarified juice into the third vessel and drawing off the concentrated syrup from the first, or hottest one, where crystallization does not take place. An American evaporator, called the Lillie, is so constructed that it can be worked forwards as well as backwards by merely turning a few cocks and valves, so that highly concentrated syrup, or even smooth-boiled masse cuites, may be obtained from the hottest vessel, which can, if desired, be heated by steam under pressure. Objections have been raised to this mode of working, on account of the formation of incrustation on the tubes, and the risk of injuring the latter when removing the scale. A further inconvenience lies in the difficulty of knowing the exact striking point when the syrup is concentrated in a closed vessel, so that sometimes it may be discharged too thin or, at other times, in a charred state.

**Miller's Finishing Battery.**

This latter inconvenience is avoided in Miller's finishing battery in which syrup concentrated in the usual way is evaporated almost to dryness.

The battery consists of a series of jacketed pans, standing over a furnace. The jackets are filled with paraffin oil having a high boiling point, which transmits the heat of the fire to the concentrated syrup in such a way that the latter boils regularly without danger of being overheated or charred. The final point is found by taking up a portion of the boiling sugar from the pan and cooling it by blowing upon it, when, if sufficiently concentrated, the sample hardens and becomes brittle. The exact point is important, for if the sugar is underboiled, it may not dry properly in the trays; and if overboiled, it is of fine grain and does not break up so easily. The pan is fitted with a discharge door, so that the charge is run out quickly and is led directly into a drying tray, where it is spread evenly with a spade, and is pushed backwards and forwards over the

tray until it thickens, which happens in about three minutes. After 15 minutes the sugar crystals will have formed and absorbed the molasses and on being turned over and raked the whole becomes changed into a soft, yellow crystalline powder free from lumps. A fresh charge of syrup is poured into the pan and the operation is repeated. As the pan has a jacket, its contents are not exposed to direct fire, and there is no danger of charring, even if the pan remains for a little while without a fresh charge, as would be the case if directly suspended over the fire.

**Kestner's Evaporator.**

It is also possible to evaporate the syrup very rapidly in a Kestner single-effet evaporator, where syrup of 50 Brix can be concentrated to 2 per cent. of water in a few minutes by superheated steam at 135° C. (275° F.) and afterwards cooled to about 100° C. (212° F.). This process is so rapid that during the short time the syrup is exposed to the high temperature no coloration or charring occurs.

CHAPTER IV.

# CURING.

## I.—Curing of First Sugar.

The operations now to be described have for their object the mechanical separation of the masse cuite into crystallized sugar and molasses. It is evident

**Principle.**

that the most advantageous way to effect this separation is that in which the maximum of crystallized sugar is obtained at once as first product, because if portions of it become again mixed with the molasses, they can only be recovered after much trouble and expense (and not even then entirely), in the less valuable after-products. It has been proved that the curing is most easily effected, and with a minimum of loss, when the sugar crystals are well-developed, and the mother-liquor limpid and free from so-called "false" grain.

All the preceding operations have co-operated to this end. The mills extracted as much juice as possible from the raw material, clarification removed all matters liable to make the juice turbid, a moderate quantity of lime threw down the pectine and albumen, avoiding the formation of viscous, dark coloured lime-salts, after which filtration rid the juice of all suspended impurities. Concentration *in vacuo* prevented overheating and decomposition of sugar, and, finally, care was taken during the boiling process to form large regular crystals, free from false grain. Notwithstanding this, careless working after the masse cuite leaves the pan may nullify all the advantages thus gained. When discharged from the pan, the hot masse cuite contains more sugar in solution than it can hold after it has been cooled down. In masse cuites containing much

**Crystallization on Cooling.**

mother-liquor, as in the case of syrup masse cuites which have been diluted with molasses, the quantity of dissolved sugar is much greater than in undiluted masse cuites. Not only is the amount of liquid larger, but owing to the slow crystallization of sucrose from impure liquids all the sugar capable of crystallizing has not yet assumed that state in the pan.

We have already seen that the sugar crystals are in constant movement in the pan and come into contact with all parts of the liquid from which

**Crystallization at Rest.**

the sugar is crystallizing out; the crystals growing in size by sugar being deposited on them instead of forming new crystals. When such motion ceases owing to the masse cuite being discharged into crystallizing tanks, or in cases where the masse cuite is very stiff and no circulation exists, the sugar afterwards crystallizing out will assume the form of

false grain. With pure masse cuites, containing but little false grain, the influence is not perceptible, but from impure masse cuites (especially when boiled at a high temperature), a great deal of sugar will crystallize out afterwards and, even if the boiling is carefully conducted, will cause the cooled masse cuite to contain so much false grain that trouble and loss will arise on curing it.

This is especially the case with masse cuites boiled very close and afterwards diluted with a large quantity of hot molasses; if these be cooled down without certain precautions a mass would result that could not be separated into crystals and molasses. Pure masse cuites can safely be cooled down in the crystallizers and then give a maximum yield on curing, but very impure ones should either be cured hot (in which case the sugar crystallizing afterwards cannot interfere with the curing) or they should be cooled in motion, which operation is indispensable for masse cuites mixed with molasses.

When first molasses are to be mixed with a subsequent masse cuite, the dissolved sugar will find opportunity to crystallize in the pan and it is therefore superfluous to waste time and cooling space. Those masse cuites may be cured hot, but as there is some danger of forming false grain when the whole of a masse cuite cannot be cured off at once, and becomes cooled, it is advisable to provide the masse cuite tank with a stirring apparatus, or to discharge the masse cuites into a large tank in which an Archimedian screw revolves, which keeps them in motion.

**Crystallization in Motion.**

When, however, it is desired to obtain exhausted molasses from the cured masse cuites, it is necessary to cool and stir previous to curing, so as to get all the dissolved sugar to crystallize in such a form that it may be easily recovered, that is to say to induce it to deposit on the crystals already present. To this end, it is necessary to maintain a gentle movement and circulation throughout the whole mass, which, at the same time, is allowed to cool gradually. This so called crystallization in motion does not increase the quantity of sugar crystallizing out (as has been repeatedly asserted), but imparts to the newly crystallizing sugar the same form as that which crystallized in the pan, and therefore yields a mixture of homogeneous sugar crystals and a limpid molasses which can be separated rapidly and without loss.

**Apparatus for Crystallization in Motion.**

The apparatus for crystallization in motion may be divided into two classes, viz., open and closed. Both consist of cylindrical vessels, sometimes provided with a "jacket" into which hot or cold water can be introduced, and fitted with a shaft with dashers arranged in spiral form, which, by revolving, keep the contents in regular and gentle motion.

The open vessels are uncovered, and the masse cuite runs into them through an open trough. On the other hand, the closed vessels have an air-tight connection with the discharge outlet of the pan and with a mixer placed above the centrifugals, and are worked under a vacuum. An air-compressor enables

them to be discharged by means of compressed air. When the highly concentrated masse cuite, diluted with the necessary amount of hot molasses, is to be transferred from the pan to the crystallizer, steam is turned off from the pan, the air-cock opened, also the discharge valve, and air pumped out from the crystallizer. The diluted masse cuite is drawn into the latter with great rapidity, when the pan is again ready for use. The crystallizer being now closed, the masse cuite is slowly cooled in constant motion, after which compressed air is admitted and the discharge door opened, causing the cold masse cuite to pass into the mixer of the centrifugals. In the open systems, the masse cuite runs through an open gutter into the vessel, is stirred, and afterwards discharged into a suction-tank, from which it is conveyed to the centrifugals by means of a chain-pump or an elevator. Before the masse cuite is run into the crystallizer, the apparatus should be heated in order to prevent the hot masse cuite from depositing false grain on coming in contact with the cold plates. The shaft should already be in motion for, if started when the vessel is full, there is risk of breakage. During the stirring and cooling, the masse cuite becomes harder, and frequently more hot molasses have to be added in order to prevent the dashers breaking. After some time, water is introduced into the jacket (in such apparatus as are furnished with one), and it is necessary to allow the air to escape from the jacket through a vent-cock, as otherwise the cooling is very irregular. In order to promote rapid cooling, the inner walls of the jackets must be occasionally cleaned to remove sediments deposited from the cooling water.

**Progress of Crystallization during Cooling in Motion.**

The following tables shows the gradual decrease in sucrose content in the mother-liquor during the cooling process. A little masse cuite was taken from the crystallizer from time to time, the molasses separated by filtration, and analysed in the usual way.

I. Composition of the masse cuite when discharged from the pan:—

Brix, 93·96; Pol., 68·4; Purity, 72·8.

| Composition of Mother-Liquor. | Temperature in °C. | Brix. | Pol. | Purity. | Per cent. Crystals in m.c. |
|---|---|---|---|---|---|
| After 1 hour's cooling .. .. .. .. | 65 | 90·36 | 40·1 | 44·3 | 48·13 |
| ,, 3 hours' cooling .. .. .. .. | 63 | 90·0 | 39·4 | 43·76 | 48·50 |
| ,, 5 ,, .. .. .. .. | 61 | 89·76 | 39·0 | 43·4 | 48·79 |
| ,, 7 ,, .. .. .. .. | 60·5 | 89·62 | 38·6 | 43·0 | 49·15 |
| ,, 9 ,, .. .. .. .. | 59·5 | 89·76 | 38·2 | 42·57 | 49·44 |
| ,, 11 ,, .. .. .. .. | 58 | 89·76 | 38·0 | 42·33 | 49·54 |
| ,, 13 ,, .. .. .. .. | 57 | 89·84 | 37·6 | 41·85 | 50·0 |
| ,, 15 ,, .. .. .. .. | 56 | 90·0 | 37·7 | 41·89 | 50·10 |
| ,, 17 ,, .. .. .. .. | 56 | 90·0 | 37·2 | 41·33 | 50·29 |

II. Composition of the masse cuite, Br. 91·32, Pol. 68·4, Purity 74·9.

| Composition of Mother-Liquor. | Temperature in °C. | Brix. | Pol. | Purity. | Per cent. Crystal in m.c. |
|---|---|---|---|---|---|
| Before cooling | 69 | 87·86 | 43·0 | 49·0 | 46·38 |
| After 2 hours' cooling | 67·5 | 87·6 | 41·6 | 47·49 | 47·66 |
| ,, 4 ,, | 66 | 87·3 | 41·0 | 46·97 | 48·02 |
| ,, 6 ,, | 64 | 87·96 | 40·2 | 45·0 | 49·77 |
| ,, 8 ,, | 62·5 | 87·62 | 39·9 | 45·5 | 49·27 |
| ,, 12 ,, | 60 | 87·62 | 38·8 | 44·28 | 50·12 |
| ,, 16 ,, | 57 | 87·62 | 38·2 | 43·6 | 50·49 |
| ,, 20 ,, | 54·5 | 86·9 | 37·2 | 42·8 | 51·22 |
| ,, 24 ,, | 52 | 87·76 | 37·0 | 42·17 | 51·67 |
| ,, 26 ,, | 51·5 | 87·76 | 36·6 | 41·7 | 52·01 |
| Separated molasses | — | 83·56 | 36·4 | 43·58 | 50·51 |

III. Composition of the masse cuite, Br. 97·70, Pol. 61·80, Purity 63·2.

| Composition of Mother-Liquor. | Temperature in °C. | Brix. | Pol. | Purity. | Per cent. Crystal in m.c. |
|---|---|---|---|---|---|
| Before cooling | 70 | 94·1 | 35·80 | 38·0 | 40·5 |
| After 6 hours' cooling | 60 | 93·7 | 33·83 | 36·1 | 42·2 |
| ,, 12 ,, | 52 | 93·6 | 32·50 | 34·7 | 43·4 |
| ,, 18 ,, | 47 | 93·2 | 31·33 | 33·6 | 44·3 |
| ,, 24 ,, | 44 | 93·1 | 30·73 | 33·0 | 44·8 |
| ,, 30 ,, | 30 | 93·0 | 30·30 | 32·5 | 45·1 |
| Separated molasses | — | — | — | 34·2 | — |

IV. Composition of the masse cuite, Br. 97·70, Pol. 61·30, Purity 62·7.

| Composition of Mother-Liquor. | Temperature in °C. | Brix. | Pol. | Purity. | Per cent. Crystal in m.c. |
|---|---|---|---|---|---|
| Before cooling | 71 | 95·4 | 35·2 | 36·8 | 40·2 |
| After 8 hours' cooling | 61 | 94·1 | 30·9 | 32·8 | 43·9 |
| ,, 16 ,, | 53 | 92·1 | 28·7 | 31·1 | 45·7 |
| ,, 24 ,, | 45 | 91·7 | 27·0 | 29·4 | 46·9 |
| Separated molasses | — | — | — | 33·3 | — |

It will be seen that crystallization progresses continuously during cooling, the increase in crystals per cent. masse cuite being respectively 2·16, 5·63, 4·6, and

6·7, on 52, 53·5, 59·5, and 59·8 per cent. of mother-liquor originally present, corresponding to an increase in crystals of 4·1, 10·5, 7·7, and 11·1 per cent. on molasses. As these figures are calculated from the degrees Brix and polarization they do not strictly represent the dry substance and sucrose. As the actual figures are given in the tables on pages 233 and 234, together with the apparent values, and as the decrease in purity during cooling is the same as in the above instances, we make use of those figures in the table given here.

| Constituents. | I. | | II. | | III. | | IV. | |
|---|---|---|---|---|---|---|---|---|
| | Last Sample. | Molasses. | Last Sample. | Molasses. | Last Sample. | Molasses. | Last Sample. | Molasses. |
| Dry substance .. .. .. .. .. | 82·4 | 82·0 | 84·7 | 83·2 | 85·3 | 80·0 | 84·0 | 83·2 |
| Sucrose .. .. .. .. .. .. .... | 40·3 | 31·8 | 37·3 | 31·6 | 38·8 | 32·0 | 38·7 | 33·6 |
| Water .. .. .. .. .. .. .. .. | 17·6 | 18·0 | 15·3 | 16·8 | 14·7 | 20·0 | 16·0 | 16·8 |
| Actual purity .. .. .. .. .... | 48·9 | 38·8 | 44·0 | 38·0 | 45·5 | 40·0 | 46·1 | 40·4 |
| Apparent purity .. .. .. .. .. | 38·4 | 28·3 | 33·7 | 27·0 | 33·4 | 27·5 | 32·8 | 27·5 |
| Sucrose on 100 water .. .. .... | 229 | 177 | 244 | 188 | 264 | 161 | 242 | 194 |
| Decrease in purity (actual) .. .. .. | 10·1 | | 6·0 | | 5·5 | | 6·5 | |
| Decrease in purity (apparent) .. .... | 10·1 | | 6·7 | | 5·9 | | 5·3 | |
| Calculated sucrose crystallized out .. | 13·6 | | 8·2 | | 7·8 | | 8·0 | |
| Decrease in sucrose on 100 water .... | 52 | | 56 | | 103 | | 48 | |

About 9 per cent. sucrose on 100 parts of molasses have, therefore, crystallized out during cooling, which greatly reduces the amount of sugar dissolved in 100 parts of water. According to the figures for dry substances, masse cuites Nos. I., II., and IV. have not been diluted with water or molasses during cooling, but No. III. has undoubtedly received an addition of diluted molasses, as seen from its originally high concentration. The ratio of sucrose on 100 of water is consequently affected, and does not represent the effect of after-crystallization. Attention may be drawn to the fact that, notwithstanding such dilution, the decrease in purity is not less than in the other cases, so that dilution has not caused sucrose to be dissolved, and has not even hindered its crystallization. As we shall see later, this dilution might be carried further without risk of diminishing the after-crystallization, and this strange phenomenon will then be explained.

**Effect of Addition of diluted Molasses to the cold Masse Cuite.**

**Solubility of Sucrose in the Water of the Molasses.**

The solubility of pure sucrose in pure water is as 300 : 100 at 64° C. and at 28° C. 215 : 100, or a decrease of 85. In the example cited above the decrease is not so great, being 52, 56, and 48 respectively, omitting the diluted No. III., which again shows that the ratio of sucrose to water in the molasses bears no relation to the solubility of pure sucrose in pure water.

**Rate at which the Temperature falls during Cooling.**

The temperature falls regularly during the cooling process, but of course more rapidly at first. In crystallizers provided with jackets, the contents cool more rapidly than in those which cool by contact with the air, but as the cooling must proceed slowly in order to obtain a regular crystallization, and be continued down to the desired temperature, nothing is gained by accelerating or retarding the rate of cooling.

Sax* mentions that in open crystallizers, without jackets, the temperature of the contents decreased 14-15° C. in the first 12 hours in some factories, and 10-12° C. in others, while in a jacketed crystallizer the decrease amounted to 2° C. per hour. It is evident that when the difference in the temperature between the masse cuite and the atmosphere or cooling-water is greatest, as at the outset, the decrease in temperature per unit of time is also greatest.

**Mixing Masse Cuites with Diluted Molasses.**

In many cases the masse cuite is so concentrated (100° Brix = 94 per cent. dry substance) that the stirring apparatus fails to keep the mass in motion, and dilution becomes necessary, as also for the subsequent curing operation. Having previously noted that a very high concentration of the masse cuite is indispensable for a maximum yield of crystals and a well exhausted mother liquor, it seems strange that such a mass can be diluted with impunity, yet a masse cuite concentrated to 100° Brix can safely be diluted to 96° Brix without dissolving the crystallized sugar. The highly concentrated masse cuite loses so much combined water, (*i.e.*, water of hydrated combinations of salts and sugar) that the first effect of dilution is to restore this combined water, and only when this is effected can fresh additions of water dissolve sugar. Of course, the dilution is not carried to this latter stage in practice, but stops before the saline combinations are fully hydrated. Such "additions of water" cannot be made by merely pouring water upon the concentrated masse cuite, because these would not mix and the water would then dissolve some of the whole masse cuite instead of only diluting the concentrated mother-liquor throughout the whole bulk of masse cuite.

The water is therefore added to the masse cuite either through a perforated pipe in the bottom of the crystallizer, or hot diluted molasses of 80° Brix are poured on the top of the masse cuite. The hot molasses readily mixes with the highly concentrated molasses surrounding the crystals, so that the mixture becomes more fluid and in proper condition for the subsequent curing in the centrifugals.

In example III., on page 248, where molasses were diluted to 86 Brix, the purity of the molasses obtained on curing did not rise above 27·3, while, in another instance when a masse cuite was concentrated to 99·5° Brix and diluted with hot molasses in the crystallizers, the resulting molasses gave 86·1° Brix and 26·8 apparent purity. A comparison of the ratios of ash to reducing sugar in the

* *Archief voor de Java Suikerindustrie*, 1899, 246.

masse cuite and the molasses showed that the low quotient of purity was really due to crystallization and not to decomposition or inversion.

Although it is not possible to state limits which will apply in every case, we may say that a highly concentrated mixed masse cuite may be diluted down to 96° Brix, the limit of dilution allowed will not then be attained, still less exceeded.

**Curing in Centrifugals.**

The separation of crystals from the molasses is effected in centrifugal drums with perforated walls, which are made to revolve at a high speed. The masse cuite is poured into the open top of the drum, which is then set in motion, whereby the masse cuite is forced against the perforated walls of the drums which retain the crystals, but allow the molasses to percolate through. Around the revolving drum is an iron casing, in which the molasses are caught and from which they escape to a gutter.

**Pug-mill.**

Masse cuite cooled in any apparatus for crystallization in motion is ready for curing, but a masse cuite cooled at rest must first be broken up into loose crystals in a pug-mill, where it is mixed with molasses and reduced to a homogeneous mass—thus obviating the risk of charging the centrifugals irregularly.

The way in which a masse cuite is boiled and cooled has considerable influence on the yield from the centrifugals.

**Shape of the Sugar Crystal an important Factor.**

When the crystals are regular and well formed, and the molasses limpid and not turbid from minute crystals, the separation is effected very easily and without loss. But if the crystals are irregular in size, or if the masse cuite contains false grain, a considerable part of the crystallized sugar passes away with the molasses and is lost as first product. The minute crystals form an emulsion with the molasses, which firmly adheres to the large crystals and can only be removed by a copious application of water. Apart from the minute crystals which escape with the molasses through the holes of the centrifugal gauze, the surfaces of the larger crystals are dissolved in the water and increase the loss. Finally, the molasses obtained are thereby diluted and can re-dissolve sugar from fresh quantities of masse cuite with which it is mixed in the pug-mill or mixer.

**Composition of First Masse Cuites and the Sugar Crystals contained therein.**

In the *Sugar Cane*, 1895, 309, the author published a list of analyses of first masse cuites, together with those of the mother liquors occurring around the crystals, the percentage of crystallized sugar and of sugar actually obtained on curing.

The masse cuites were boiled from syrup without addition of molasses, and cooled in the stationary tanks in general use at that time. The masse cuite was dug out from the tanks and conveyed to the pug-mill in wooden barrels, which

occasioned some trifling mechanical loss; but a more serious loss occurred during curing. Besides the minute crystals, which pass through the interstices of the centrifugal gauze, sugar is lost by being dissolved in the water or syrup added during curing, or in the diluted molasses added to the pug-mill. But, whereas the former (mechanical) losses are real, these last are not so, since, although the the sugar is not recovered in a solid and saleable form, it is still present in the molasses; from which it may be recovered, but only with much cost and labour, and then not completely. In any case, the yield of sugar as first product is diminished and the main object of the operation is thereby rendered less effective.

| No. | Mode of Manufacture. | Masse Cuite. | | | | | | | Syrup. | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Sucrose. *a* | Reducing Sugar. *b* | Ash. *c* | Water. *d* | Actual Quot. of Purity. *e* | Brix. *f* | Apparent Quot of Purity. *g* | Sucrose. *h* | Reducing Sugar. *i* | Ash. *j* | Water. *k* | Actual Quot. of Purity. *l* |
| 1 | Washed with syrup .. .. .. | 84·8 | 2·74 | 2·25 | 8·23 | 91·3 | 95·4 | 88·9 | 59·2 | 7·29 | 6·07 | 25·12 | 79·1 |
| 2 | Boiled as usual .. .. .... | 84·2 | 3·20 | 1·37 | 9·77 | 93·4 | 93·3 | 90·3 | 58·7 | 9·97 | 3·82 | 25·71 | 82·3 |
| 3 | Boiled very strong .. .. .. | 86·7 | 3·20 | 1·39 | 6·15 | 92·4 | 96 0 | 90·3 | 50·9 | 11·09 | 4·80 | 21·23 | 64·6 |
| 4 | Centrifugalled warm .. .... | 74·4 | 10·77 | 1·59 | 9·03 | 81·8 | 95·0 | 78·5 | 37·7 | 26·76 | 3·86 | 22·13 | 48·3 |
| 5 | Centrifugalled cold .. .. .. | 74·1 | 11·07 | 1·54 | 9·02 | 81·4 | 94·9 | 78·1 | 32·9 | 28·59 | 3·91 | 23·37 | 42·9 |
| 6 | Carbonated juice .. .. .... | 86·7 | 1·83 | 1·90 | 7·58 | 93·9 | 92·8 | 93·4 | 61·1 | 5·41 | 5·79 | 22·19 | 78·5 |
| 7 | Ordinary mode of manufacture | 78·9 | 8·99 | 1·16 | 7·76 | 85·3 | 94·4 | 83·6 | 33·7 | 28·12 | 3·65 | 24·50 | 44·6 |
| 8 | Ordinary mode of manufacture | 79· | 8·47 | 1·15 | 7·82 | 85·7 | 94·3 | 83·7 | 32·9 | 27·53 | 3·72 | 26·12 | 44·7 |
| 9 | Centrifugalled warm.. .. .. | 83·6 | 4·82 | 1·40 | 7·97 | 90·1 | 95·1 | 87·9 | 52·7 | 14·01 | 4·03 | 23·01 | 68·4 |
| 10 | Centrifugalled cold .. .... | 84·1 | 4·81 | 1·40 | 8·11 | 91·3 | 95·0 | 88·6 | 50·2 | 15·14 | 4·35 | 25·31 | 67·2 |
| 11 | Boiled very soft .. .. .. .. | 79·6 | 6·72 | 1·01 | 11·41 | 89·9 | 91·2 | 87·3 | 49·0 | 16·52 | 2·52 | 27·77 | 67·8 |
| 12 | Boiled soft.. .. .. .. .... | 80·4 | 6·81 | 1·03 | 10·11 | 89·4 | 92·5 | 87·2 | 42·9 | 19·93 | 3·00 | 28·93 | 60·4 |
| 13 | Boiled as usual .. .. .. .. | 81·1 | 6·93 | 1·12 | 9·66 | 89·8 | 92·8 | 87·2 | 43·1 | 20·87 | 3·39 | 28·45 | 60·1 |

| No. | Mode of Manufacture. | Sugar. | | | | | Crystals present in 100 Masse Cuite. *r* | Crystals yielded by 100 Masse Cuite. *s* | Wet Sugar yielded by 100 Masse Cuite *t* | Sugar lost on 100 Masse Cuite. *u* | Sugar lost on 100 Crystals. *v* | Washed with water in c.c. per cwt. *w* | Number of holes per sq. c.m. of gauze. *x* |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Sucrose. *m* | Reducing Sugar. *n* | Ash. *o* | Water. *p* | Crystals in 100 Sugar. *q* | | | | | | | |
| 1 | Washed with syrup .. .. .. | 96·7 | 0·54 | 0·43 | 1·56 | 93·14 | 62·93 | 59·46 | 63·94 | 3·47 | 5·51 | 500 Syrup 25° Bé | 81 |
| 2 | Boiled as usual .. .. .... | 96·4 | 0·63 | 0·32 | 2·18 | 91·86 | 64·13 | 62·01 | 67·5 | 2·12 | 3·31 | 400 | 80 |
| 3 | Boiled very strong .. .. .. | 97·9 | 0·38 | 0·13 | 0·60 | 97·27 | 71·05 | 62·15 | 63·9 | 8·90 | 12·51 | 1200 | 80 |
| 4 | Centrifugalled warm .. .... | 95·1 | 1·92 | 0·20 | 2·28 | 93·69 | 58·83 | 51·34 | 54·8 | 7·49 | 12·73 | 2000 | 100 |
| 5 | Centrifugalled cold .. .. .. | 95·8 | 1·12 | 0·16 | 2·52 | 94·35 | 61·62 | 51·51 | 54·6 | 10·11 | 16·40 | 2800 | 100 |
| 6 | Carbonated juice .. .. .... | 96·2 | 0·48 | 0·57 | 1·68 | 90·15 | 67·10 | 66·80 | 74·1 | 0·30 | 0·45 | .. | 100 |
| 7 | Ordinary mode of manufacture | 94·1 | 1·60 | 0·27 | 2·54 | 91·46 | 68·22 | 58·81 | 64·3 | 9·41 | 13·79 | 3000 | 100 |
| 8 | Ordinary mode of manufacture | 96·8 | 0·64 | 0·22 | 1·62 | 94·02 | 69·09 | 55·28 | 58·8 | 13·81 | 19·88 | 800 | 81 |
| 9 | Centrifugalled warm.. .. .. | 98·2 | 0·37 | 0·12 | 0·78 | 96·93 | 65·26 | 58·40 | 60·25 | 6·76 | 10·36 | 400 | 81 |
| 10 | Centrifugalled cold .. .... | 98·5 | 0·51 | 0·16 | 0·26 | 96·99 | 67·81 | 64·10 | 66·1 | 3·71 | 5·47 | Steam | 81 |
| 11 | Boiled very soft.. .. .. .. | 95·5 | 1·35 | 0·25 | 2·30 | 90·54 | 59 92 | 45·27 | 50· | 14·20 | 23·66 | 700 | 100 |
| 12 | Boiled soft.. .. .. .. .... | 95·7 | 1·27 | 0·14 | 2·06 | 94·63 | 65·67 | 55·44 | 58·58 | 10·23 | 15·58 | 500 | 100 |
| 13 | Boiled as usual .. .. .. .. | 96·8 | 1·01 | 0·14 | 1·60 | 95·45 | 66·96 | 60·0 | 62·85 | 6·96 | 10·38 | 300 | 100 |

From the tables on page 251 it will be seen that the quantity of crystallized sugar in masse cuite, containing different percentages of water, but boiled from the same syrup, increases with the concentration, but that the yield on curing does not always follow this rule.

**Influence of Water Content of Masse Cuites on Centrifugal Yield.**

In the table given below, Nos. 2 and 3 are masse cuites from the same syrup, but concentrated to 9·77 and 6·15 per cent. of water respectively; Nos. 11, 12, and 13 are also boiled from the same syrup, and contain 11·41, 10·11, and 9·66 per cent. of water respectively. The figures for crystallized sugar and crystals obtained on curing are calculated for a water content of the masse cuite of 9·77 in Nos. 2 and 3, and for a water-content of 9·66 in Nos. 11, 12, and 13, in order to simplify the calculation.

| No. | Degree of Concentration. | Crystallized Sugar in Masse Cuites. | Crystals obtained on Curing. | Raw Sugar obtained on Curing. | Sugar Lost. | | Washed with Water in c.c. per cwt. |
|---|---|---|---|---|---|---|---|
| | | | | | Per 100 Masse Cuite. | Per 100 Crystals in Masse Cuite. | |
| 2 | Usual concentration.. .. | 64·13 | 62·01 | 67·5 | 2·12 | 3·31 | 400 |
| 3 | Very heavily concentrated. | 68·31 | 59·75 | 61·43 | 8·52 | 12·13 | 1200 |
| 11 | Very slightly concentrated | 61·02 | 46·17 | 51·— | 14·48 | 24·13 | 700 |
| 12 | Slightly concentrated .... | 66·05 | 55·72 | 58·87 | 10·28 | 15·64 | 500 |
| 13 | Usual concentration.. .. | 66·96 | 60·— | 62·85 | 6·96 | 10·38 | 300 |

Two other masse cuites were divided into two parts, one of which was cured hot and the other after cooling for a week in waggons or in tanks.

| No. | | Crystallized Sugar. | Crystals obtained | Raw Sugar obtained. | Sugar Lost. | | Washed with water in c.c. per cwt. |
|---|---|---|---|---|---|---|---|
| | | | | | Per 100 m.c. | Per 100 Crystals. | |
| 4 | Cured warm .. .. .. | 58·83 | 51·34 | 54·80 | 7·49 | 12·73 | 2000 |
| 5 | ,, cold .. .. .... | 61·62 | 51·51 | 54·60 | 10·11 | 16·40 | 2800 |
| 9 | ,, warm .. .. .. | 65·62 | 58·40 | 60·25 | 6·76 | 10·36 | 400 |
| 10 | ,, cold .. .. .... | 67·81 | 64·10 | 66·10 | 3·71 | 5·47 | Steam |

When discharged from the pan, a crystallized masse cuite contains a certain quantity of dissolved sugar in the form of syrup, and which crystallizes out on cooling. The cold masse cuite, therefore, contains more crystallized sugar, and the enclosed syrup proportionally less dissolved sugar, than when the masse cuite left the pan. But the sugar which crystallizes out on cooling is not always

obtained on curing, as is shown in Nos. 4 and 5, in which the yield of crystals and of raw sugar is the same before and after cooling, although the masse cuite contained about 3 per cent. more crystals after cooling than before.

Whether this reduced yield is due to the fine grain passing through the liners of the centrifugals, or to the greater viscosity of the cold molasses necessitating a free application of purging-water, it is impossible to say, but probably the loss of crystals may be attributed to both causes jointly. As regards the increased viscosity of the molasses after cooling, it may be mentioned that the masse cuite cured cold required 800 cubic centimetres more purging-water per cwt. than the same masse cuite when cured hot, but still yielded the same quantity of cured sugar.

The much purer masse cuites, Nos. 9 and 10, gave quite different results ; again, more sugar crystallized out from the cooled masse cuite, but this deposited upon the crystals already formed, causing the syrup to remain liquid and capable of being easily removed. The warm masse cuite required 400 cubic centimetres of purging-water per cwt. whilst on curing the cold one a small quantity of steam admitted into the outer casing sufficed to obtain fairly white sugar. The possibility of obtaining the sugar dissolved in the hot syrup, but which crystallizes on cooling, depends therefore on the shape in which this sugar crystallizes out. When in the form of very minute crystals, the yield may be even less than if the masse cuite had been cured warm, although more crystals have been formed.

The experiments show that the yield depends not only on the quantity of sugar present in the masse cuite, nor yet on the quantity of crystals it contains, but to a large extent too on the size and form of the sugar crystals. Taking this into consideration, it becomes evident why the loss of crystals in the experiments 11, 12, and 13 increases with the dilution of the masse cuite. The masse cuite in No. 11 contained 50 per cent. of mother-liquor, which would not remain in contact with the crystals during cooling, but floated above them. The water-content of the mixture was about 15 per cent. The sugar which was present in a super-saturated state, and that, also, which is more soluble at 60° than at 30° C. crystallized out as false grain in a thick impure liquid, and out of contact with the sugar crystals already formed. The consequence was a viscous molasses requiring much purging-water, and a consequent loss of crystals.

In No. 12, the water-content was less, and consequently more sugar had crystallized out in the pan, and less in the crystallization tanks, so that the yield was better. A still higher yield was obtained from the more concentrated No. 13, in which not only had more sugar crystallized, but in such a form that it could be recovered. But concentration has its limits, as is shown by close boiled masse cuites Nos. 2 and 3, possessing a higher percentage of crystals, but yielding less than a more dilute masse cuite boiled from the same syrup. This lower yield was undoubtedly caused by the viscosity of the molasses, which necessitated a freer application of purging-water to remove it from the crystals during curing.

We noticed that if the masse cuite contains a large amount of mother-liquor and crystallization is allowed to take place at rest, there is great risk of the formation of false grain. But when such a masse cuite is cooled in motion, the sugar crystallizes upon the crystals already formed, increasing their size and not forming fresh crystals which are lost in the centrifugals. The crystals obtained are of uniform size, and can be easily separated from the fluid molasses.

**Crystallization in Motion.**

With pure masse cuites the difference between cooling at rest and in motion is small, as more sugar crystallizes in the pan and less on cooling, and because the latter has a tendency to form larger crystals than is the case with impure syrups. As modern methods involve the introduction of molasses or syrups into the pan in one way or another, after-crystallization of first masse cuites is becoming customary. Even if first masse cuites are cured direct, it is still advisable to keep them in motion before curing, because if this latter operation be postponed owing to some accident, and the masse cuites be allowed to cool at rest, an opportunity for the formation of false grain is given with all the inconvenience attached thereto.

A good example of the influence of cooling in motion on the form of the crystals, and hence upon the yield obtained on curing, is afforded by the following analyses of four masse cuites, boiled in the same way from rather impure syrups, but of which three were cooled at rest, and one in motion.

| Cooling. | Masse Cuite. | | | | | Molasses. | | | | | Sugar Crystallized. | Amount of Crystals obtained on Curing. | Loss of Crystals on Curing per 100 of Sugar. |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Sucrose. | Glucose. | Ash. | Water. | Quotient of Purity. | Sucrose. | Glucose. | Ash. | Water. | Quotient of Purity. | | | |
| At rest .. .. | 74·1 | 11·07 | 1·54 | 9·02 | 81·44 | 32·9 | 28·59 | 3·91 | 23·37 | 42·86 | 61·62 | 51·51 | 16·40 |
| ,, .. ... | 78·9 | 8·99 | 1·16 | 7·76 | 85·32 | 33·7 | 28·12 | 3·65 | 25·40 | 44·63 | 68·22 | 58·81 | 13·79 |
| ,, .. .. | 79·0 | 8·47 | 1·15 | 7·82 | 85·72 | 32·9 | 27·52 | 3·72 | 26·12 | 44·67 | 69·09 | 55·28 | 19·88 |
| In movement.. | 77·0 | 9·62 | 2·51 | 7·49 | 83·45 | 35·3 | 26·54 | 6·70 | 22·94 | 46·40 | 64· | 64· | — |

It will be seen that the quantity of crystallized sugar did not differ much in all four cases, neither did the chemical analyses of the masse cuites or of the molasses, but there are marked differences in the quantity of sugar obtained on curing. While all the crystallized sugar in the masse cuite cooled in motion could be obtained on curing, in the other cases, where the after-crystallization had taken place at rest, so much false grain had been formed that no less than from 13 to 20 per cent. of the crystallized sugar re-dissolved. This table therefore shows the great influence of the shape of the crystals on the yield obtained, and how important it is to pay close attention to the boiling of first sugar, where, by want of care, more loss can be suffered than at any other stage of the

manufacture. Apart from improving the shape of the crystals, crystallization in motion has the further advantage of being a cleanly operation and free from mechanical losses.

**Difficulties met with in Curing.**

The curing of first masse cuites, boiled without addition of molasses and derived from pure juice, does not present any difficulties, but it sometimes happens that the molasses present in the masse cuite are so gummy that they do not permeate the crystalline layer, but remain inside, and cannot be removed by washing. The only remedy is to stop the centrifugal after it has run for a certain time, mix the sugar with the layer of molasses, and then centrifugal again, but as the loosened mass does not distribute itself evenly against the walls of the drum, this device is not very satisfactory.

Sometimes masse cuites are so stiff that the curing takes too long, and one bad strike of masse cuite retards the whole curing plant for hours, and occasions a stoppage in the factory routine. In such a case, it should be ascertained that the centrifugals are driven the prescribed number of revolutions, *e.g.*, 1200 per minute, as (owing to the slipping of the belts) a reduction of a couple of hundred revolutions per minute may remain unnoticed, but is sufficient to retard the curing.

It is also desirable to steam the centrifugal liners once every day and thus keep the meshes clear of sugar crystals which are apt to choke them.

**Regulation of the Curing Work.**

Assuming that the centrifugal plant is in good order, one centrifugal is charged with the masse cuite which has to be cured. If this sample cures without any difficulty, the whole of the masse cuite is transferred to the mixer of the centrifugals, and cured. But, if the trial sample cures badly, the masse cuite is mixed with hot molasses and another trial made If this shows no improvement, the masse cuite may be added in small portions to larger quantities of easily cured masse cuite and the resulting mixture cured with as little delay as possible. But if the quantity be too large to mix with other masse cuites, it is better to store it in a tank for a fortnight, as the most obstinate masse cuite is more easily cured after such a period of rest.

**Sugar Crystals in the Molasses Gutter.**

Sometimes sugar crystals are found in the molasses gutter, which are much too large to have passed through the mesh of the centrifugal liners, and which indicate that the liners have been torn or otherwise damaged. Of course, it is impossible to obtain exhausted molasses with such a defect, however much care and trouble be bestowed on the boiling and cooling. It is well to point out this source of loss here, and to advise the daily inspection of the centrifugal liners so as to ensure replacing the damaged ones before too much crystallized sugar has passed through them.

**Purging.**

It is not feasible to separate all the molasses from the sugar crystals in the centrifugals and, consequently, the crystals remain covered with a thin film of molasses after that operation. When making refining crystals from pure syrup masse cuites, this film consists of neutral molasses of 60-70 degrees

purity, and, being harmless, it will not give rise to sourness or deterioration. But when making white, or raw sugar from masse cuites to which molasses have been added, the adhering film of molasses is of low purity and may cause sourness, so that it is necessary to remove this by pouring water on the sugar while the centrifugal is in motion. The water dilutes the molasses which then escape through the perforated walls of the drum. This separation is called purging, covering, or washing, and must be performed very cautiously in order to avoid dissolving too much sugar at the same time. Usually, the water is sprayed upon the sugar whilst the centrifugal is in full motion, after allowing sufficient time for the molasses to pass out of the drum, leaving the sugar fairly dry. Owing to the rapid motion, every portion of the sugar receives its share of the water, which, after passing through the crystals, escapes through the liner. Various appliances have been devised to spread the water in a fine spray over the sugar in the drum, and so to moisten a large surface with a small quantity of water, or a nearly saturated solution of less valuable sugar is used for covering, which, while it removes the molasses, cannot dissolve any crystallized sugar. Finally, steam is used for covering, or a jet of steam which carries air along with it; the steam becomes condensed on the crystals, forming water, which dissolves the thin layer of molasses, and is separated with this.

However carefully this work be done, it is impossible to prevent some sugar being dissolved during purging; even when purging with sugar syrup much pure sugar will pass over into the molasses. When curing raw sugar, the molasses from which are to be returned to the pan, this dissolving of sugar is of minor importance, because it returns to the pan in a product of the same composition as that from which it came. But when curing a masse cuite yielding exhausted molasses, any dissolving of the sugar in the purging-water is to be avoided, since this would raise the sucrose-content and purity of this waste product, and result in a palpable loss of sugar. Similarly, when making white sugar, the mixing of so-called "green" molasses with the purging-syrup is irrational, because much water or steam is required to remove the last trace of colour from the crystals, and consequently so much sugar is dissolved that a large amount of covering syrup is obtained, having a higher purity than the original syrup, and which, when mixed with the molasses, reduces the yield of first sugar considerably.

**Separation of Green Molasses and Covering Syrup.**

When curing first masse cuites for white sugar and mixed masse cuites yielding exhausted molasses, it is necessary to keep the molasses quite separate from the purging-water, and to collect them in separate receptacles. This has the further advantage that one may use an unlimited amount of purging-water, because the sugar dissolved therein returns to the pan in the form of a product of the same purity as that from which the sugar was derived, instead of passing into an inferior product (molasses).

The simplest way of performing this separation is to provide two gutters behind the centrifugals, one for the molasses proper, the other for the purer washings. A movable outlet from the centrifugal discharges the molasses into that gutter where it belongs by simply inclining it to one side or the other. This, however, is not quite sufficient, as the viscid molasses has not entirely left the outer drum when purging has started, so that a partial mixing of the two in the outer drum cannot be avoided. Moreover, the outlet is not shifted automatically, and there is consequently risk of this operation being sometimes omitted. Another method is an arrangement in the outer casing of the centrifugal, which necessitates the centrifugal being able to rotate in opposite directions by means of an alteration in the driving-gear.

A third device is a centrifugal having two casings, one inside the other; each of which is provided with a discharge outlet. One casing is fixed, as in the ordinary type, while the inner casing may be shifted up or down by means of a lever. Whilst the true molasses are being separated, the movable casing is raised, so that the molasses are caught in the fixed one, and pass through its discharge pipe into the molasses receiver. As soon as purging commences the movable casing is lowered, and catches the purging syrup, which escapes through the other discharge pipe into a separate receiver.

All these appliances are very ingenious and work well when properly handled, but there is a risk that the workmen will sometimes omit to shift the levers when purging begins or ends. So long as there is a chance of doing something wrong, nothing will prevent careless workmen from doing so occasionally. When experiments or trials have been made with such apparatus they always worked admirably, but in practical working it will occasionally happen that the rich purgings will get into the receptacle intended for molasses or *vice versa*. The first eventuality is the more serious, because if the rich purgings are once mixed with the exhausted molasses they are lost, and the loss of sucrose in waste molasses is unnecessarily increased.

**Curing in two sets of Centrifugals.**

To be on the safe side, the curing may be carried on in two sets of centrifugals. In the first, the poor molasses are separated, and the raw sugar then mixed with purging-syrup in a pug mill, and again cured in the second set of centrifugals. The fluid obtained from this second curing operation is partially used for mixing with the raw sugar, while the remainder is returned to the juice. Usually, the purity of such washings is superior to that of the syrup from which the first masse cuites are boiled, and there is therefore no objection to their being returned to the clarified juice. It is not advisable to return them to the syrup, because, being of higher density, they might trouble the pan boiler who prefers to work with syrup of uniform density, especially when boiling masse cuite for white sugar. But when the purging-syrup is returned to the clarified juice, it passes through the

évaporating plant, and a uniform syrup is supplied to the vacuum pans without danger of loss of sugar.

Instead of employing two sets of centrifugals, it has been proposed to separate the true molasses in a very large centrifugal of special construction and open at bottom. The masse cuite enters it from the mixer when the centrifugal is running at full speed, the sugar clinging to the walls of the basket, while the molasses pass through. By slackening the speeed of the machine, so as to reduce the centrifugal force, the sugar is caused to slip down until it falls through the open base of the basket into a second pug-mill, where it is mixed with purging-syrup and then finally cured in the ordinary centrifugals.

**"Blueing" of Sugar.**

When the sugar is quite cured and purged, it is sometimes "blued" in order to neutralize the last traces of yellow colour. Sometimes an emulsion of ultramarine in water is drawn into the vacuum pan at the graining point in order that the nucleus of each crystal may contain sufficient blue to neutralize the yellowish tint of the crystals when fully grown. In other cases, a little of a similar dilute emulsion is poured into the centrifugal after the purging water is separated. The finely divided ultramarine remains between the crystals imparting to them a bluish tint which pleases the eye of the purchaser. It is advisable to mix the ultramarine with water in a suitable mixer in order to break up any lumps which could cause blue spots in the sugar instead of a diffused tint. Some employ aniline blue or indanthrene, but ultramarine is the most universal dye for white sugar in refineries, as also in factories where white sugar is made directly from cane juice.

**Lump Sugar.**

Instead of sugar in loose crystals, some factories make sugar in lumps, resembling the lump sugar of the refineries.

This class of sugar is made from fine-grained masse cuites, intended for white sugar, and cured as usual. The moist white sugar is mixed with pure syrup and treated in special centrifugals, having small boxes attached to the interior of the drums, the bottoms of which consists of centrifugal gauze. The mixture of crystals and syrup fills the boxes, the syrup escapes, and the sugar is purged with moist steam, which causes the crystals to stick together in a hard lump. As soon as the purging-syrup is colourless, the sugar is discharged in the form of square white lumps which now only need drying. The trade mark of the factory is generally stamped on the gauze bottom of the boxes, so that every lump of sugar is moulded with this mark, without requiring any special apparatus for the purpose.

In the same way, slabs of sugar may be made which are cut into cubes by means of automatic scissors.

We noted that white sugars are cured twice, and that raw sugars, from masse cuites which yield rich molasses, may be purged in the centrifugals without

**What to do with the so called Molasses Sugar.**

trouble. It now remains to state what is done with the sugar cured from second or third masse cuites which yield exhausted molasses. It is evident that purging would, in this case, cause irreparable loss of sugar, so that curing separates the masse cuites into exhausted molasses, and a moist sugar containing a considerable quantity of the same exhausted molasses. In a former chapter, we explained that in order to obtain a well exhausted molasses, the crystals in these last masse cuites ought to be small, so as to present a sufficient surface whereon fresh sugar may deposit when assuming the crystallized state. If complete exhaustion of the molasses is aimed at, we have now to deal with a very fine-grained sugar, coated with exhausted molasses, and the problem is how to dispose of them to the best profit.

When there is a demand for such sugar, the problem is solved by selling it, but in many cases such low grade sugar is not wanted, and the manufacturers have to work it up in some way or other.

**Mixing with first Masse Cuites in the Coolers.**

In cases where a fine grained first sugar is not objected to, the most rational and economic plan is to transform this low-grade sugar into first sugar, by boiling the first masse cuites to fine grain and mixing this with the low-grade sugar in the crystallizers. The first masse cuites contain crystals and a mother-liquor of about 50 purity and, when mixed with low-grade sugar (consisting of crystals and exhausted molasses) the crystallized sugar from both sources is recovered as a uniform grain, and a mixed molasses separates, the purity of which will be higher than 30 but below 50. This mixing has the same effect as if the low-grade sugar were mixed with rich syrup in a mingler before being cured a second time, and this operation is therefore equivalent to curing in two sets of centrifugals. After being well mixed, the mixture of masse cuite and sugar is cured, the sugar is purged with water, syrup, or steam, and the resulting washings allowed to mix with the molasses, because these will be reboiled to form a second masse cuite. In this way, the low-grade sugar is got rid of without undergoing any loss of the adhering exhausted molasses, the latter being replaced by a much purer molasses, which, if necessary, may be removed by purging-water, because the portion of sugar dissolving during that operation is recovered in the form of a product of the same purity as the original masse cuite.

It is evident that the purity of the rich mother-liquor, surrounding the crystals of the first masse cuite, will be reduced by being mixed with the exhausted molasses introduced in the form of low-grade sugar, and this reduction in purity depends on the purity and quantity of low-grade sugar added.

Starting from a pure juice, the quantity of low-grade sugar will be much less than in the case of an impure juice, and, consequently, the decrease in purity will be much larger in the second case than in the first.

Let us assume an example in which a first masse cuite of 80 purity yields molasses for a second mixed masse cuite of 70 purity, the molasses from which are

re-boiled to a molasses masse cuite of 60 purity. We can then calculate how far the purity of the mother-liquors deteriorates by the return of the low-grade sugars to the first masse cuites.

We shall assume that in one case a low-grade sugar of 90 polarization is returned and in another case one of 85 polarization and that they are returned into a masse cuite of 80 purity or into one of 70 purity, giving one example of each.

Sugar having 99° Brix and 90 polarization (yielded by a masse cuite containing mother liquor of 30° purity) shows a purity of $\frac{90 \times 100}{99} = 91$ and contains $\frac{91-30}{100-30} \times \frac{99}{100} = 86$ per cent. crystallized sucrose and 14 per cent. molasses. 100 parts of molasses masse cuites of 60 purity yield $\frac{60-30}{91-30} \times \frac{95}{99} = 47{\cdot}2$ parts of moist low-grade sugar, which contain $\frac{47{\cdot}2 \times 14}{100} = 6{\cdot}6$ parts of exhausted molasses.

A sugar having 98 Brix and 85 polarization shows a quotient of 86·7, and contains 79·4 per cent. of crystallized sucrose and 20·6 per cent. of molasses, while 100 parts of molasses masse cuites yield 50 parts of moist low-grade sugar containing 10·3 parts of exhausted molasses.

According to Pasma's figures,* and working by scheme B, we have on every 100 parts of molasses masse cuite 130 parts of masse cuite at 70, and 650 at 80. When all the molasses sugar is returned into the masse cuite at 70, then, in case of a sugar of 90 polarization, 6·6 parts of exhausted molasses are mixed with 130 parts of a masse cuite of 70, or in 100 parts $\frac{6{\cdot}6 \times 100}{130} = 5{\cdot}1$. (For simplying the calculation the water contents of the different masse cuites are assumed to be the same.) We assume all the masse cuites to be 95 Brix, and all the molasses to be 90 Brix and that, according to Pasma, a masse cuite of 70 purity gives molasses of 50 purity, and one of 80 purity gives a molasses of 60 purity.

100 parts of masse cuite at 70 purity contain $\frac{70-50}{100-50} \times \frac{95}{100} = 38$ parts of crystals, and 62 parts of mother-liquor of 50. To these are added 5·1 parts exhausted molasses at 30 purity, causing the purity to decrease as follows:—

$$\begin{array}{rr} 62{\cdot}0 \times 0{\cdot}50 = & 31{\cdot}00 \\ 5{\cdot}1 \times 0{\cdot}30 = & 1{\cdot}53 \\ \hline 67{\cdot}1 & 32{\cdot}53 \end{array}$$

48·5 or a decrease of 1·5

If the low-grade sugar had polarized 85, $\frac{10{\cdot}3 \times 100}{130} = 7{\cdot}9$ parts of exhausted

* See page 237. *Archief voor de Java Suikerindustrie*, 1904, 614.

molasses would have been mixed with the 62 parts of mother-liquor bringing down the purity by 2·3 degrees as here shown:—

$$\begin{array}{rr} 62{\cdot}0 \times 0{\cdot}50 = & 31{\cdot}00 \\ 7{\cdot}9 \times 0{\cdot}30 = & 2{\cdot}37 \\ \hline 69{\cdot}9 & 33{\cdot}37 \end{array}$$

47·7 or a decrease of 2·3

If, on the contrary, the low-grade sugar had been mixed with masse cuite of 80 purity, the decrease would have been much less, owing to the considerable quantity of that masse cuite.

On every 100 parts of molasses masse cuite, we have not less than 650 masse cuite of 80 purity. 100 parts of this contain $\frac{80-60}{100-60} \times \frac{95}{100} = 47{\cdot}5$ parts of crystals, or 52·5 parts of molasses at 60.

6·6 parts of exhausted molasses are added to 650 parts of first masse cuite, or on 100 parts, $\frac{6{\cdot}6 \times 100}{650} = 1$ part in one case and $\frac{7{\cdot}9 \times 100}{650} = 1{\cdot}2$ parts in the other case. The decrease in purity in the case of sugar of 90 becomes $60 - \frac{52{\cdot}5 \times 0{\cdot}60 + 1 \times 0{\cdot}30}{53{\cdot}5} = 0{\cdot}5$ and in the case of sugar of 85 $60 - \frac{52{\cdot}5 \times 0{\cdot}60 + 1{\cdot}2 \times 0{\cdot}30}{53{\cdot}7} = 0{\cdot}7$.

When calculating these figures for other purities of the juice, we obtain the following table for the decrease in purity of the mother-liquor in the first and second masse cuites, when all the low-grade sugars are returned to them.

| | Quotient of Purity of the Syrup. | | | | |
|---|---|---|---|---|---|
| | 90 | 88 | 85 | 82 | 75 |
| Parts of 1st m.c. on 100 molasses m.c. .. .. .. .. .. | 650 | 451 | 463 | 162 | 56 |
| ,, 2nd ,, 100 ,, .. .. .. .. .. | 130 | 127 | 123 | 118 | 118 |
| *When returning sugar of 90 Pol.* | | | | | |
| Parts of waste molasses on 100 parts of 1st m.c. .. . | 1·0 | 1·4 | 1·4 | 4·1 | 11·8 |
| ,, ,, ,, 100 ,, 2nd ,, .. .. . | 5·1 | 5·2 | 5·2 | 5·6 | 5·6 |
| Decrease in quotient of mother-liquor 1st m.c. .. .. . | 0·5 | 0·8 | 0·8 | 2·4 | 4·2 |
| ,, ,, ,, ,, 2nd ,, .. .. . | 1·5 | 1·6 | 1·6 | 1·6 | 1·6 |
| *When returning sugar of 85 pol.* | | | | | |
| Parts of waste molasses on 100 parts of 1st m.c. .. .. . | 1·6 | 2·3 | 2·2 | 6·4 | 18·4 |
| ,, ,, ,, 100 ,, 2nd ,, .. .. . | 7·9 | 8·1 | 8·4 | 8·8 | 8·8 |
| Decrease in quotient of mother-liquor 1st m.c. .. .. . | 0·7 | 1·3 | 1·2 | 3·5 | 5·8 |
| ,, ,, ,, ,, 2nd m.c. .. .. . | 2·3 | 2·3 | 2 4 | 2·5 | 2·5 |

We notice that, when returning low-grade sugar into second masse cuite, the purity of the mother-liquor does not decrease more when the original purity of the syrup is high, than when it is low.

On the contrary, this purity exerts a great influence on the quantity of low-grade sugar obtained, which rises considerably when the purity of the syrup falls. This is clearly shown in the following table, where all the figures are calculated on 100 parts of dry substance in syrup.

| | Quotient of Purity of the Syrup. | | | | |
|---|---|---|---|---|---|
| | 90 | 88 | 85 | 82 | 75 |
| Total amount of masse cuites .. .. | 176 | 160 | 155 | 158 | 151·5 |
| Quantity of molasses—masse cuite .. | 20 | 25·4 | 33 | 42 | 60 |
| ,, sugar of 90 .. .. .. | 9·4 | 12·– | 15·6 | 19·8 | 28·3 |
| ,, ,, 85 .. .. .... | 10 | 12·7 | 16·5 | 21 | 30 |

Whereas, at a high purity of the syrup, the amount of low-grade sugar to be returned is not excessive and will not cause any trouble, it increases considerably when the purity of the original syrup falls below 85. Under these conditions, it is not wise to return all this sugar into first or second masse cuites, since by doing so the centrifugal work will be increased by 25 per cent.

When, therefore, the juice is impure, and consequently the amount of low-grade sugar becomes excessive, it often happens that the centrifugals fail to do the required work properly, and that the low-grade sugar still contains an excessive quantity of molasses owing to its being imperfectly cured. By doing so, much more exhausted molasses is returned to the first masse cuites than the calculated quantities, so that the purity of the mother-liquors in such masse cuites decreases much more than might have been expected. In such cases, when it is seen that the amount of low-grade sugar is getting too large to be properly dealt with, it is best not to force matters, but to cure only so much masse cuite as the centrifugal plant can deal with properly, and to store the remainder in tanks until an opportunity of curing it occurs.

**Use of Low-grade Sugar as Seed for First Masse Cuites.**

This mixing of low-grade sugar into first masse cuites can only be done when the size of the grain is of no importance. To obtain a well exhausted molasses it is essential that the grain of the low-grade sugar be fine. In order to mix this with first masse cuites, it is evident that the grain of this ought to be fine also, otherwise the mixed sugar will contain small and large crystals, and will, moreover, give rise to many difficulties in refining operations.

When a large-grained first sugar is desired, such a mixture is no longer feasible, and the low-grade sugar may then be used as seed in boiling these masse cuites, as follows :—The sugar, well cured in the centrifugals (but without purging), is mixed with syrup and the mixture drawn into the pan and concentrated. The syrup washes off the adhering molasses leaving the sugar crystals clean and brilliant, just as when graining in the ordinary way. The quantity of grain thus added must be sufficient (as when graining syrup) to yield the requisite number of fully grown crystals in the finished masse cuite. The small crystals are allowed to grow by the addition and concentration of more syrup, and the masse cuite finished in the usual way. A great advantage of this method is that the hot syrup dissolves all the smaller grains from the added sugar, so that ultimately the seed consists exclusively of well-shaped crystals.

**Drying the Sugar.**

White sugar, from which the molasses have been entirely removed by washing, may be dried in the centrifugals by a jet of dry steam. When the sugar is discharged after that operation, it only requires passing through a coarse sieve before being packed perfectly dry. The raw sugars which are still coated with an adhering layer of moist molasses cannot be thus dried; they are not dried at all in some countries, but are shipped with a tolerably high moisture content, while in other countries no sugar is shipped without having previously undergone a drying process.

Formerly, the sugar was dried in the sun on a plastered floor, but, owing to the increased output of factories, this method has been generally abandoned, the more so because it was expensive, slow, and dependent on the weather. The impossibility of drying sugar on rainy days was not so serious then as now, because wet weather also interfered with the transport of canes and the drying of bagasse, so that all manufacturing operations stopped. Sugar driers came into use as soon as railway transport and modern bagasse furnaces rendered the manufacturer quite independent of the weather and enabled him to continue crushing even during rainy weather.

**Sugar Driers.**

At the present day, the sugar is dried in large iron cylinders, placed on an incline, and provided internally with scoops. The moist sugar is thrown in at the top, and the revolving motion of the cylinder causes it to fall from one scoop on to another until it reaches an outlet at the bottom, after remaining in the dryer for 7-10 minutes. During that time, a current of hot air is driven in, or drawn through the dryer, the air being heated by being first drawn through a series of pipes built into a furnace under which bagasse is burnt.

On entering the dryer, the hot air comes into contact with the falling moist sugar and deprives it of moisture.

Usually the hot air enters the dryer at a temperature of 120° C. (248° F.) and with a water content of 20 grms. per cubic metre, and leaves it at a temperature of

50° C. (122° F.) with a water content of 40 grms. per cubic metre. As air, at that temperature, is only saturated when containing 50 grms. of water per cubic metre, it is evident that this drying process is not as economical as it might be. The temperature of the dried sugar is also about 50° C., the refining crystals then contain 0·50 per cent. of moisture and the white sugar not more than 0·1 per cent.

In some systems, the drying is effected with steam, or steam is conducted through heating tubes by means of a current of air, but those in which the sugar is dried by means of hot air are most generally used.

**Dullness of Sugar when Dried in Revolving Dryers.**

Sugar crystals retain their brilliancy when dried by means of dry steam in the centrifugals, or in the sun, though in the latter case they are somewhat bleached, but they become dull and grey after passing through the hot-air dryer; the commercial value of the sugar being thereby depreciated. This dullness is probably due to the rubbing of the sugar crystals against each other and against the metallic parts of the dryer; also to the finely powdered sugar arising from the crystals already dried and carried forward by the air current, and deposited upon the moist crystals entering at top. In the case of white sugar, which ought to have a sparkling appearance, the drying should be effected in the centrifugal by means of dry steam.

**Dried Sugar sometimes becomes Moist again.**

Sometimes dried sugar absorbs moisture from the atmosphere after being passed through the dryer, and forms into a hard mass. This is observed when diseased or burnt canes have been worked up, and also when the sugar has been exposed to too high a temperature in the dryer. It may be remedied by passing the sugar through a second apparatus, similar to the dryer, through which a current of cold dry air is drawn. If sugar is bagged hot, it often sets into a hard brown mass, which, being very hygroscopic, readily attracts moisture from the atmosphere, so that a syrupy liquid oozes out of the bags. When white sugar is bagged hot, it becomes dark-coloured in the centre of the bags after being stored for some time. These inconveniences are avoided by cooling in a second cylinder.

**Sifting and Packing.**

After being dried, the sugar is passed through copper strainers in order to remove lumps formed in the vacuum pans or centrifugals, or pieces of uncured masse cuite which have dropped into the centrifugal after the sugar was cured. The finished sugar is then packed in bags, baskets, or casks.

## II.—Treatment of Low-Grade Products.

The proportion of second sugar, per 100 parts of first sugar, depends entirely on the quality of the latter. We saw that when making refining crystals, by the method described on page 229 and following, all the available sugar can be recovered as first product to the exclusion of any seconds. This is feasible even when making white sugar, though with more trouble and skill; but generally a larger or smaller quantity of sugar remains as low-grade product. On the other hand, if market prices allow, it is better to mix the first molasses with syrup and boil this mixture entirely to seconds, thus greatly increasing the yield of that sugar. This, however, is an exception, and as a rule first and second sugars are made in the ratio of 5 : 1 unless one or other of the "absolute recovery" processes is adopted.

**Quantity of Low-Grade Product Obtained.**

As the first molasses are generally diluted in the factory with water or the condensed steam used for purging, they are in a very favourable condition for fermentation, and should therefore be concentrated as quickly as possible. As molasses contain all the impurities from the masse cuite in a much more concentrated state than in the juice, a special clarification of the molasses is sometimes necessary.

**Clarification of the Molasses.**

The molasses also contain suspended impurities, which ought to be removed before a new crystallization takes place, otherwise they may be occluded by the crystals and impart to them a dark colour. If, on the contrary, they remain suspended in the mother-liquor after crystallization has taken place, they are apt to render it viscous and difficult to separate from the crystals. Finally, molasses obtained from badly boiled masse cuite, or one which has been cured hot, may contain very minute crystals, due either to false grain which has passed through the liners, or to after-crystallization of the hot molasses in the gutters or tanks. These ought to be re-dissolved before the molasses are evaporated again. If returned into first masse cuite, such small crystals will give rise to irregular crystallization even if the masse cuite itself has been carefully boiled. When the molasses are boiled smooth, the crystals settle on the coils and become charred, and also cause an irregular crystallization by their being mixed with the crystals which form during cooling. Strictly speaking, we should never omit to re-clarify all molasses before boiling them. To this end they are run into an eliminating pan, and heated by means of a steam jet. A froth now appears on the surface which contains all the floating and suspended impurities of the molasses; this is skimmed off. At the same time the steam, and the resulting dilution, dissolves the fine crystals which had passed through the interstices of the centrifugal liners, and were suspended in the molasses.

The analyses of the froth and of the molasses from which it is derived are given below, both in their original state and also calculated on dry substance so as to eliminate the influence of the dilution.

| Constituents. | Molasses. | | Froth. | |
|---|---|---|---|---|
| | On 100 Original Molasses. | On 100 Dry Substance. | On 100 Original Froth. | On 100 Dry Substance. |
| Sucrose | 47·21 | 71·02 | 49·50 | 68·78 |
| Reducing sugar | 9·80 | 14·74 | 9·26 | 13·06 |
| Water | 33·53 | — | 28·03 | — |
| Ash | 1·98 | 2·90 | 3·25 | 4·57 |
| Soluble part of the ash | 1·23 | 1·85 | 1·18 | 1·67 |
| Insoluble part of the ash | 0·75 | 1·14 | 2·07 | 2·90 |
| Silica | 0·16 | 0·24 | 0·56 | 0·78 |
| Calcium phosphate | 0·08 | 0·12 | 0·38 | 0·53 |
| Calcium carbonate | 0·40 | 0·60 | 0·96 | 1·35 |
| Magnesium carbonate | 0·11 | 0·17 | 0·17 | 0·32 |
| Wax | — | — | 1·57 | 2·20 |

It follows that the froth chiefly consists of the same constituents as the molasses, but is rendered gummy by small admixtures of phosphate and carbonate of lime, wax, and organic salts of lime.

This admixture is similar in composition to the precipitate obtained during defecation and to the various sediments encountered during the course of manufacture. It occurs in the defecation mud, on the copper gauze serving to filter the clarified juice, in the first vessels of the evaporating plant, in the sediment from the syrup, in the scale of the vacuum pan, and now again in the froth of the molasses. This shows how desirable it is to eliminate the scums after clarification so that subsequent operations need not be hindered by having to repeat this operation at every stage of manufacture, which causes much annoyance and delay. If this turbid molasses be diluted to about 60° Brix and treated in the special centrifugal, referred to on page 229, or in a suitable filter, the gummy suspended matters could be separated, leaving the molasses limpid. This would be specially advantageous before returning them into a syrup masse cuite which is intended to yield exhausted molasses, because the mother-liquor being very fluid, crystallization would be rapid and the molasses would be easily separated from the crystals in the centrifugals, while the sugar would contain a minimum of molasses when added to a first masse cuite. This clarification is also recommended when molasses are reboiled for the last time without being mixed with a syrup masse cuite. It is true that it does not raise the quotient of purity, and that the effect of clarification cannot be expressed in figures, but the elimination of the constituents which render the molasses viscous is a greater advantage than an increase of one or two points in purity, since such increase is generally more apparent than real.

During elimination, molasses can be neutralized with lime, if necessary, or may be bleached (when white seconds are to be made) by means of sulphurous acid or hydrosulphite, just as in the case of syrup. Molasses from carbonated juice can even be filtered after sulphitation, while those from defecated juice are generally too viscous to pass through cloth, but may be filtered through palm fibre or similar material, though but slowly.

**Sulphitation of the Molasses.**

First molasses, such as are obtained when making white sugar, can be boiled to grain. This is done in the pan with steam chamber and stirring apparatus, described in a previous chapter. As the second sugars must have a very fine grain, the pan is grained when nearly full so that the whole heating surface is covered. After that, the contents of the pan are concentrated as in the case of first masse cuites, the grain being allowed to grow at the expense of the rich molasses, and subsequently inferior molasses are drawn in and the boiling finished in the usual way. The essential thing in this process is that the purity of the molasses be sufficiently high to allow of their being grained, which is only possible with very superior molasses.

**Boiling Molasses to Grain.**

When white sugar is made, the first molasses can easily be grained in the pan and cooled in motion, which is an excellent method of separating the first molasses into sugar and exhausted molasses in one operation.

We must bear in mind that it is not possible to exhaust these final molasses to the same extent as when the first sugar is made from mixed masse cuites. The purity of a first molasses masse cuite is quite as low as that of a mixed masse cuite of syrup and molasses, but the latter has been prepared by adding molasses to the already crystallized syrup masse cuite, in which well developed crystals were already present, so that the sugar crystallizing out from the comparatively impure liquid was therefore deposited upon these crystals. When graining the molasses, new crystals have to be formed, which takes some time. Moreover, they do not grow very large and their growth is sluggish, since sugar crystallizes but slowly from impure liquids. We do not obtain large and well shaped crystals and a limpid mother-liquor as in the former case, but a thick magma of very fine crystals saturated with molasses. Consequently, the evaporation cannot proceed as far as in the case of first masse cuites, and we are compelled to leave more water and consequently more sucrose in the molasses than in the former case.

**Crystallization in Motion.**

The finished masse cuite is discharged into crystallizers and cooled in motion, but as the fine grain does not subside rapidly, it is not necessary to stir constantly; it is quite sufficient to rotate the cooler for an hour, thrice in 24 hours. After two or three days the cooled second masse cuite can be cured.

The following examples refer to molasses which were diluted, steamed, skimmed, boiled to grain, and cooled in motion. When cured, the sugar was

purged with water, for which reason the purity of the molasses is in most cases higher than that of the mother liquor in the masse cuite.

Molasses of 66·7 purity were grained, highly concentrated, and cooled in motion during 2 × 24 hours. The analysis of the masse cuite gave Brix 96; polarization 64·0; reducing sugar 11·1; purity 66·7. The composition of the mother-liquor during the different phases of cooling was as follows:—

| Sample taken. | Temperature in °C. | Brix. | Polarization. | Reducing Sugar. | Quotient. | Sucrose Crystallized. |
|---|---|---|---|---|---|---|
| When discharged .. .... | 73· | 93·2 | 40·0 | 18·2 | 42·9 | 40· |
| After 12 hours cooling .. | 65·5 | 91·9 | 35·6 | 19·2 | 38·7 | 43·8 |
| ,, 48 ,, ,, ... | 51· | 91·4 | 32·8 | 20·2 | 35·9 | 46·1 |
| Molasses obtained on curing | — | 85·8 | 33·6 | 19·4 | 39·2 | 43·4 |

This masse cuite was very stiff and had to be diluted with water, and yet the molasses adhered so firmly to the sugar that a good deal of water was required in purging, causing the purity of the resulting molasses to rise excessively.

A second molasses was boiled in the same way but less concentrated; it was, however, stirred for a longer time and cooled to a lower temperature.

Analysis of the masse cuite: Brix 92·6, Pol. 59·2, Reduc. sugar 12·25, Quot. 63·9.

| Sample taken. | Temperature. | Brix. | Pol. | Reducing Sugar. | Quotient. | Sucrose Crystallized. |
|---|---|---|---|---|---|---|
| On striking .. | .. 72 .. | 91·3 .. | 45·0 .. | 16·12 .. | 49·28 .. | 25·6 |
| After 24 hours | .... 60 .. | 89·7 .. | 36·8 .. | 18·31 .. | 41·02 .. | 35·2 |
| ,, 48 ,, | .. 51 .. | 88·9 .. | 34·0 .. | 19·22 .. | 38·24 .. | 37·0 |
| ,, 72 ,, | .... 44 .. | 88·6 .. | 32·8 .. | 20·83 .. | 37·02 .. | 38·9 |
| ,, 96 ,, | .. 39·5 .. | 88·3 .. | 31·2 .. | 21·27 .. | 35·33 .. | 39·8 |
| Separated molasses .. | — .. | 87·4 .. | 34·0 .. | 19·84 .. | 38·90 .. | 38·0 |

This table shows that the crystallization during cooling follows the same course as was noticed in the case of first masse cuites.

The less pure molasses are not boiled to grain but to "string-proof." They are drawn into the pan until all the coils are covered, concentrated, and molasses added in proportion as the contents diminish through evaporation, and concentrated to "string-proof" as soon as the pan is quite full. The thread should break sooner in the case of second masse cuites than in thirds, &c., although the cooled mass should be sufficiently fluid in order to promote crystallization and not become sufficiently solid to retard crystallization. Great care must also be taken that the concentrated second masse cuite does not cool too rapidly, nor commence to crystallize before it enters the crystallizing tanks. The temperature should therefore be kept rather high during the finishing stage, and not fall below 70° C. (158° F.) This high temperature, however, has the disadvantage that the

**Boiling Smooth.**

unstable products of decomposition which become accumulated in the seconds, attain such a degree of concentration that they may decompose spontaneously, and when once this has started it cannot be stopped, as will be shown under the heading "froth fermentation." But, although a high temperature is sometimes destructive, it is necessary in order to prevent a too rapid crystallization of the sugar.

**Cooling slowly at Rest.**

When a second masse cuite, boiled smooth, deposits crystals on its way to the crystallizing tanks, there is great risk of this sugar interfering with the regular crystallization of the masse cuite and the formation of well developed crystals. These very minute crystals do not grow, but render the mother-liquor (in the cooled masse cuite) viscous and turbid and interfere with the curing, causing considerable loss. In some factories, the concentrated molasses flow into the crystallizing tanks through long gutters, or, still worse, through pipes; conditions which offer every opportunity for irregular crystallization and its attendant evils. For this reason, it is advisable to convey the low-grade masse cuites to the crystallizing tanks with as little delay as possible, preferably in trolley-tanks which can be moved rapidly. The author has inspected many factories where the yield from second masse cuites was usually very low, and in such cases a rather rich molasses was sent away from the factory, because it would no longer yield any sugar. These wasteful practices disappeared as if by magic when these masse cuites were no longer allowed to flow slowly through pipes to the crystallizing tanks, but were carried there rapidly in trolley-tanks.

**Cooling in Motion.**

At one time second masse cuites, boiled smooth, were cooled in motion, sometimes after addition of sugar crystals as seed, but the results did not seem satisfactory and the practice was abandoned.

An example of a concentrated second masse cuite, boiled smooth, and cooled for three days in motion is recorded here. The analyses are of the mother-liquor surrounding the crystals, and consequently the first one represents the composition of the original masse cuite before crystallization had commenced.

| Sample taken. | Temperature in °C. | Brix. | Polarization. | Reducing Sugar. | Quotient. | Sucrose Crystallized out. |
|---|---|---|---|---|---|---|
| Before cooling .. .. .. | 70 | 91·1 | 60·8 | 9·25 | 66·7 | — |
| After 24 hours.. .. .... | 56 | 86·8 | 46·8 | 11·02 | 53·9 | 25·3 |
| After 48 hours .. .. .. | 47 | 86·0 | 45·2 | 12·82 | 52·5 | 27·3 |
| Separated molasses.. .... | — | 85·9 | 42·8 | 13·92 | 49·8 | 30·6 |

It is not improbable that the new Kestner apparatus (in which molasses can be concentrated to about 2 per cent. of water in a very short time and at a high

temperature) will yield masse cuites which, when cooled in motion, will yield exhausted molasses and finely grained second sugar, but the experiments in that direction are not yet completed.

When concentrating slowly, and at a relatively low temperature, as is now done, molasses cannot be concentrated to the same degree as masse cuites boiled to grain, because they would become too stiff to allow of proper crystallization. We are, therefore, compelled to leave sufficient water in the low-grade masse cuite to allow crystallization to take place in the tanks, and to again concentrate the mother-liquor after separating it from the sugar which crystallizes out. This is why molasses boiled to grain can be separated into sugar and exhausted molasses (or molasses of a low purity) in one operation, whereas molasses boiled smooth are reduced to the same condition only after repeated concentrations and crystallizations.

**Cooling at Rest.**

**Composition of Raw and Concentrated Molasses.**

Although it is not possible to give figures for the composition of molasses masse cuites, the analyses of a first, second, and third molasses, before and after boiling, in the same factory, may follow here, as they admit to some extent of mutual comparison.

| | Brix. | Sucrose. | Reducing Sugar. | Ash. | Water. | Purity. |
|---|---|---|---|---|---|---|
| First molasses before boiling | 82·70 | 49·6 | 12·3 | 5·04 | 20·55 | 60·0 |
| Second ,, ,, ,, | 81·10 | 46·0 | 13·4 | 5·20 | 23·27 | 56·7 |
| Third ,, ,, ,, | 83·20 | 44·8 | 17·5 | 6·60 | 20·28 | 53·8 |
| First ,, after ,, | 89·70 | 53·6 | 12·7 | 5·45 | 16·8 | 59·8 |
| Second ,, ,, ,, | 88·40 | 49·6 | 16·0 | 5·30 | 18·3 | 56·1 |
| Third ,, ,, ,, | 90·70 | 48·5 | 17·9 | 6·87 | 14·10 | 53·4 |

In boiling molasses, not more than five or six per cent. of water is evaporated, hence the yield in sugar from molasses masse cuite cannot be very considerable if, in curing the No. 1 sugar, all the crystals were separated from the molasses.

The molasses masse cuites are discharged into crystallizing tanks, where they are allowed to cool down, and where the sugar that can now no longer be retained in solution finds opportunity to crystallize. The rapidity of this crystallization and the size of the crystals depend on the rapidity with which cooling goes on, and the nature of the liquid in which they are formed. Pure molasses masse cuites crystallize much more quickly than impure and viscous ones, when the cooling time is the same; therefore first molasses masse cuites are generally allowed to cool for a week, seconds for a fortnight, and so on, before being cured. From what has been already said, these limits need not be closely

adhered to, and an impure first-molasses masse cuite may be allowed more than a week to crystallize, if this can be done without interfering with the regular operations, and if the dimensions of the crystallizers are suitable. Cooling and crystallization proceed more rapidly in small iron tanks than in large cisterns built up from the ground.

As a rule, the tanks should be of sufficient capacity to hold the contents of one pan, and not so large as to be filled only after three or four consecutive operations, for in this case the crystallization is disturbed every time a fresh supply is run in, and the grain is irregular in form.

**Danger of excessive Concentration.**

The hot masse cuite is generally fluid, but becomes stiff and semi-solid during cooling, and as concentration is generally carried too far rather than not far enough, crystallization in the tanks is generally hindered. Crystals form, but grow so slowly or not at all, that sometimes a masse cuite, having a purity of 50°, consolidates into a hard mass which cannot be cured and is generally thrown away. It is, therefore, necessary to concentrate low-grade masse cuites to about 94° Brix, this being the limit for a good crystallization at rest. Storing of the highly concentrated second masse cuites in a hot room has been suggested for keeping the molasses fluid during crystallization, but the general construction of tropical factories would prove an obstacle in carrying out this suggestion, which has not gone beyond a few preliminary trials.

When the last masse cuites are very stiff, crystallization can be promoted by covering them with water to a depth of one foot, and leaving them to stand for a month. A few planks are first laid on the top of the masse cuite and the water poured upon them, in order to prevent the water from directly mixing with the masse cuite. The masse cuite gradually absorbs water, becoming more fluid and allowing the sucrose to form into crystals. When crystallization is complete, the water is scooped off and the masse cuite cured as usual.

**Froth Fermentation.**

During the cooling of low-grade masse cuites, the surface sometimes becomes convex and eventually bursts, a brown froth oozing out of the crevices. This soon covers the whole surface, rising continuously and finally flowing over the top of the tank. Gases escape from this froth, emitting a very unpleasant smell, and having an acid reaction, the whole phenomenon being called "froth-fermentation." With second and third molasses (in which no hard crystalline crust is formed on the surface) the whole mass starts frothing from the bottom, but in other respects the phenomena are the same as with first molasses. As this fermentation is not due to micro-organisms, all efforts to combat the phenomenon by means of disinfecting agents are fruitless. This froth fermentation does not decompose sugar, and as the masse cuite is kept in motion by the disengaged gases, crystallization takes place very easily, hence such masse cuites generally contain well-shaped crystals, and are very easily cured,

for which reason it is customary to say that the froth changes into sugar, but this must not be understood literally. The most probable hypothesis is that the frothing is caused by the spontaneous decomposition of products formed by previous decompositions of sugar or glucose, forming carbonic, formic, and acetic acids, and caramel.

Perhaps both causes may promote froth fermentation. The author recollects a very striking instance of this phenomenon in a factory where the temperature in the carbonatation tanks had been allowed to rise too high. The first sugar and first molasses masse cuites were quite normal, but suddenly two low-grade masse cuites fermented violently. They entered the crystallizing tanks at a temperature of 70° C., but after a couple of days this rose to over 100° C., and currents of acid gases escaped with such force that large lumps of the contents were sometimes flung up and fell back into the tank. Part of the masse cuite was charred by the intense heat; and, after fermentation, it appeared as a hard and porous mass of carbonaceous matter, almost insoluble in water, which extracted the following constituents:—

| | Per cent. |
|---|---|
| Sucrose .. .. .. .. .. .. .. .. .. .. .. | 7·69 |
| Reducing sugar .. .. .. .. .. .. .. .. .... | 38·4 |
| Free acid (as acetic acid) .. .. .. .. .. .. .. | 3·41 |

It has been proved that froth fermentation is also due to decomposition of products formed by overheating of sugars, and has been obviated by steaming and skimming the raw molasses, by which means the fine crystals suspended in the molasses are dissolved and can no longer become overheated by contact with the coils, and thus give rise to these troublesome products of decomposition.

A further proof that froth-fermentation is due to decomposition of products of superheating is the fact that molasses are now no longer boiled repeatedly, but almost immediately removed as exhausted, and therefore there is less risk of super-heating, with the result that froth-fermentation has occurred much less frequently of late years.

The reason why such masse cuites start frothing only while cooling down is that the unstable bodies decompose spontaneously when the concentration of the solutions reaches a certain point. As a masse cuite cools and sugar crystallizes out, the dissolved impurities become more concentrated, and this promotes decomposition, which goes on steadily in the hot medium, when once started.

When this fermentation is accompanied by a considerable rise in temperature, sucrose, glucose, fructose, &c., are also decomposed and even charred, but the decomposition generally stops at the primary products of decomposition which are already present in the liquid. It is possible that such products are always present, but that some condition which induces decomposition is absent, and no

fermentation is seen, but some local overheating, or excessive temperature, allows the fermentation to start. As it ceases when the masse cuite cools down, it is never so violent in small iron tanks, which cool rapidly, as in large ones, where the heat is retained for a longer time. As already observed, no sugar is decomposed by this fermentation, but the mechanical losses may be considerable when the mass foams over the tanks on to the floor. If this is prevented by only half filling them, the available storage space is not utilized and the capacity of the plant diminished. These are sufficient reasons for trying to prevent the fermentation by working at a low temperature during carbonatation, and by keeping defecated juices faintly alkaline to phenolphthalein paper before and during concentration. Further, all molasses should be diluted, steamed, and skimmed previous to being concentrated, and prolonged heating at temperatures above 70° C. ought to be avoided. With these precautions, froth-fermentation may be avoided.

Second molasses masse cuites of 60 purity are crystallized in about one week; those of 55 purity in two weeks, and lower grades in three weeks or more, after which they are cured. When the grain is large and the crystals do not stick together, the curing is carried out in the usual centrifugals, but such masse cuites are so fluid that they do not require to be prepared in a pug-mill. Formerly the centrifugals used for seconds were driven at a low velocity, but nowadays the seconds are cured in Weston centrifugals, which are started at a slow speed, and afterwards acquire their usual velocity.

**Curing of Seconds.**

**Drying.** The second sugar can be washed in the centrifugals if necessary, but, as a rule, this is omitted, and the moist sugar is sold, or dissolved in the juice.

The white seconds are dried in sugar dryers just as the first sugar. It is advisable to have a separate dryer, and grass-hopper conveyor for seconds, and not to dry both first and second sugar in the same apparatus, as the dryer is very difficult to clean, and lumps of second sugar may get loosened while drying white sugar and spoil the appearance of the latter. If, on the contrary, some large crystals of first sugar find their way into seconds, the buyers complain that the grain is not fine enough and is irregular. To avoid such complaints, it is better to dry both products separately so that no mixing of the two sugars can take place.

**Crushing of Seconds.** Sometimes the molasses masse cuites boiled to grain yield such large crystals that buyers refuse to accept it as second sugar. In this case the sugar may be passed through a disintegrator, a proceeding which at the same time improves the colour.

In some factories the moist white seconds are compressed into blocks, which are dried and sold for direct consumption.

When a low-grade masse cuite has a purity of 45-48, the grain is generally too small to be recovered in centrifugals, so the masse cuite is loaded into bags made of special palm-leaf mats. As soon as some molasses drain off, the bags are re-filled with the masse cuite, and this draining off and re-filling is continued until the contents have assumed a semi-solid state. The bags are then sewn up and piled in heaps, by which means a further quantity of molasses oozes out of the bags. The piles are shifted a few times, and when the contents are considered sufficiently dry the sugar is sold as "sack-sugar," "black-stroop," or "tank-bottoms."

**Sack-Sugar, Tank-Bottoms, or Black-Stroop.**

As a rule, it contains 65 per cent. of sugar crystals, and 35 per cent. of exhausted molasses.

The manufacture of this product is a very dirty and wasteful process, and any means of diminishing the quantity of masse cuite to be worked up into sack sugar would be a boon to all concerned. At first undiluted molasses flow from the bags, sometimes containing fine sugar crystals in suspension; later on a more dilute molasses drains off, moisture having been absorbed from the atmosphere but not sufficient to dissolve sugar and so raise the purity. At the end of the operation (when the bags have lain for months) the sugar also absorbs moisture, and a syrupy liquid of 50 purity drips from the bags, whereas at starting molasses of 34 purity separated out.

The probable yield of such a product cannot, of course, be calculated, and guess work is generally rewarded by complaints and claims from the manufacturers or the buyer.

If the seconds can be sold at a fair price, it is advisable to do so, but if they are only fetching a low price, or are quite unsaleable, then we are compelled to melt them in the juice, and boil them into first sugar, an operation that cannot be performed without loss, which in this case is inavoidable.

**Circumstances determine whether Sugar should be returned.**

We stated on page 270 that a low yield was obtained from second masse cuites, when the first molasses were fairly well exhausted by the improved methods of boiling and cooling. We now propose to show that a poor quality of seconds are obtained when the bulk of the sugar is recovered as firsts.

The following table shows the composition of a number of sugars intended for re-melting, all of which are taken from practical working. The percentages of crystallized sucrose and of adhering molasses are calculated from the reducing sugar content of the second sugars and the molasses separated from them.

| No. | Kind of Sugar. | Polarization. | Reducing Sugar in | | Per cent. Crystal. | Molasses. |
|---|---|---|---|---|---|---|
| | | | Sugar. | Molasses. | | |
| 1 | 1st Molasses sugar .. .. | 86·8 | 3·93 | 14·86 | 73·6 | 26·4 |
| 2 | 1st ,, ,, .... .... | 77·2 | 4·07 | 7·04 | 43·0 | 57·0 |
| 3 | 2nd ,, ,, .. .. .. | 84·8 | 5·5 | 23·9 | 77·0 | 23·0 |
| 4 | 3rd ,, ,, .... .... | 80·5 | 8·09 | 30·5 | 73·5 | 26·5 |
| 5 | 3rd ,, ,, .. .. .. | 76·7 | 9·1 | 29·0 | 68·6 | 31·4 |
| 6 | 3rd ,, ,, .... .... | 74·0 | 10·0 | 25·0 | 60·0 | 40·0 |
| 7 | 3rd ,, ,, .. .. .. | 70·3 | 10·0 | 22·9 | 56·3 | 43·7 |

When a product, containing a great percentage of exhausted molasses, is to be returned, the question arises as to which stage of the manufacture is most suitable. Some melt the second sugars in the heated mill juice, by allowing the latter to flow over a sieve on which this sugar is piled; others melt it in the subsided clarified juice, while others run off the syrup at a relatively low density and bring it to its proper concentration by dissolving the second sugar in it. All these methods have their advocates and opponents. No loss is to be feared from dissolving these sugars in the syrup, but then the mistake is made of mixing the syrup that had been carefully clarified and neutralized with the (as a rule) acid sugars, and contaminating it with their impurities. We can only do this on condition that the syrup is afterwards thoroughly eliminated, neutralized, and allowed to settle. The same must be said with regard to melting the seconds in the clarified juice; in this case also an acid and impure body is dissolved in a clarified and neutralized juice, though this is preferable to melting the same in the syrup, because they dissolve much more readily in thin juice than in the concentrated syrup. One drawback to this process is the incrustation of the tubes of the triple-effet by the suspended impurities which settle on them during concentration.

From the point of view of obtaining a good clarification, the introduction of the seconds into the mill-juice is more to be recommended, as they will then again be submitted to all the processes of clarification, against which may be set the drawbacks of the heavier juice subsiding slowly, and of the juice in the scum containing more sugar, so that more is lost in the scum-cakes.

The re-melting of seconds is usually a very wasteful operation. As there is not always an opportunity of disposing of them, they are frequently heaped up, and at the end of the grinding season, a large quantity of seconds have to be

melted in an insufficient quantity of juice, causing the last masse cuites of the season to consist almost exclusively of re-melted seconds. The waste due to re-melting is therefore a mechanical loss.

**Returning Seconds into the Vacuum Pan.**

This may be obviated by dissolving the second sugar in a little hot mill-juice to the concentration of syrup, skimming and neutralizing, and drawing it into the pan, in which a first masse cuite is already half finished. The grain already formed in this is suspended in a liquid, the purity of which is in no way superior to that of the sugar solution drawn in. In this way there is no loss, the juices are not spoiled by being mixed with inferior sugar, and no objection can be brought against this plan, because the grain is already formed, and under ordinary conditions would increase at the expense of a syrup which is no purer than the solution drawn in. A good neutralization of this sugar solution is indispensable, as otherwise the acid molasses remaining between the crystals of the sugar might cause inversion and deterioration.

Another method is to mix the seconds with syrup and to draw the mixture into the pan. Part of the sugar dissolves but the remainder serves as seed, so that not only is the syrup enriched but the duration of the boiling process is shortened by eliminating the operation of graining.

**Returning of "Black-Stroop."**

Unless the juice is well clarified (by carbonatation) or has a very high purity, it is not advisable to return the lowest grade sugars, or black-stroop, and even then this is not a wise plan. It is better to sell this product (which consists of about half its weight of molasses) even at a low price, than to run the risk of spoiling good juice through mixing it with so inferior a product.

# III.—Composition of Commercial Cane Sugars.

**Commercial Basis of Payment.**

Cane sugar is either sold on the basis of polarization, or of the "net analysis." In the former case the polariscope reading is stated without any correction for local temperature. The "net analysis" is found by subtracting the percentage of glucose (reducing sugar) and five times the percentage of ash from the polarization.

In many countries the duties on cane sugar are levied on a combined basis of polarization and colour.

**Dutch Standard.**

The Dutch Standard (D.S.) consists of a series of samples of cane sugar, ranging from a very dark No. 7 to an almost white product No. 25. They are prepared every year by two firms in Holland under the supervision of the Netherlands Trading Society (Nederlandsche Handel Maatschappij) in sealed sample bottles, which are sent to the various merchants and Customs depots. As these grades serve as standards for the different classes of sugar on which taxes are levied, it is important that sugar merchants should know exactly to which class the sugar belongs which they want to import; and, therefore, the sets of samples comprising the Dutch Standard are daily used for comparison in many countries which export cane sugar.

**Colour of the Sugar as an Indication of its Quality.**

Although colour is not a criterion of the quality of a sugar, every number of the Dutch Standard (between 11 and 17) corresponds to a certain composition. The analyses of the high grade sugars do not differ much from that of pure sucrose, the difference being mainly one of colour, rather than of composition. On the other hand, the differences in composition of the dark sugars may be so considerable that all relation between colour and analysis disappears. The average analyses of sugars between Nos. 11 and 17 D.S. are as follows:—

| Analysis. | No. 11. | No. 12. | No. 13. | No. 14. | No. 15. | No. 16. | No. 17. |
|---|---|---|---|---|---|---|---|
| Polarization .. .... | 96·10 | 96·50 | 97·10 | 97·70 | 98·30 | 98·70 | 99·0 |
| Reducing Sugar.. .. | 1·48 | 1·42 | 1·11 | 0·92 | 0·55 | 0·32 | 0·20 |
| Ash .. .. .. .... | 0·50 | 0·38 | 0·34 | 0·27 | 0·23 | 0·14 | 0·08 |
| Moisture .. .. .. | 0·82 | 0·80 | 0·82 | 0·50 | 0·33 | 0·32 | 0·25 |
| Undetermined* .... | 1·10 | 0·90 | 0·63 | 0·61 | 0·59 | 0·52 | 0·47 |
| Total .. .. .. | 100. — | 100. — | 100. — | 100. — | 100. — | 100. — | 100. — |
| Nett .... | 92·12 | 93·18 | 93·69 | 94·43 | 96·60 | 97·68 | 98·4 |

* The temperature at which these analyses are made is 28° C., or 8° C. over the temperature at which the polariscope is standardized. Therefore the polarization is about 8 × 0·003 = 0·25 lower than the real sucrose content, and consequently the figure for undetermined is too high by that amount.

The colour of a sugar is only partly due to adhering molasses, being also due to impurities incorporated within the crystals, sucrose being one of those substances which possess this property. Von Lippmann* mentions that crystals of sucrose can incorporate logwood extract and congo red, and reports that the colouring matter in sugar candy is absorbed through the crystals. Further, crystallizations of metallic compounds together with sugar are familiar, and then we know that mere traces of iron compounds and of caramel impart a dark tint to sucrose crystals formed in such solutions.

**Colour of the Crystal.**

La Bastide† mentions a case of yellow-coloured sugar obtained from syrup which had been filtered over sand containing iron. Stolle‡ studied the solubility of iron hydroxide in sugar solution, and states that, notwithstanding the fact that iron hydroxide, iron oxide, iron sulphide, and ferrous oxide are very slightly soluble, as shown in the table underneath, yet these trifling quantities were more than sufficient to impart a yellow colour to the sucrose solutions, with the exception of the solution containing iron oxide, which was coloured red.

**Coloration of Sugar by Iron Salts.**

The figures given here represent milligrammes per litre.

| Per cent. Sucrose. | $Fe_2$ (OH) 6 | | | $Fe_2$ $O_3$ | | | $Fe_3$ $O_4$ | | | Fe S | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | 17·5° | 45° | 75° | 17·5° | 45° | 75° | 17·5° | 45° | 75° | 17·5° | 45° | 75° |
| 10 | 3·4 | 3·4 | 6·1 | 1·4 | 2·0 | — | 10·3 | 10·3 | 12·4 | 3·8 | 3·8 | 5·3 |
| 30 | 2·3 | 2·7 | 3·8 | 1·4 | — | — | 12·4 | 10·3 | 12·4 | 7·4 | 1·9 | 7·2 |
| 50 | 2·3 | 1·9 | 3·4 | 0·8 | 1·1 | — | 14·5 | 10·3 | 15·5 | 9·9 | 19·8 | 9·1 |

Herzfeld repeatedly ascribed a greyish tint of the sugar to the presence of an iron compound, which remained dissolved in the juice owing to defective clarification. In former years, the author remembers seeing beetroot second products, that had assumed a greenish or dark brown tint, which examination proved to be due to an iron compound.

Every year brings fresh instances of coloration due to iron oxide, which causes the dark colour of the first syrup masse cuites and sugars when the manufacturing period opens. That this coincides with a relatively high iron-content is shown by the following instances.

* *Die Chemie der Zuckerarten*, II., 1062.
† *Archief voor de Java Suikerindustrie*, 1903, 954.
‡ *Zeitsch. f. d. Rubenzuckerind.*, 1906, 338.

A number of first masse cuites and first sugars were analysed at the commencement of a grinding season, and later on a number of similar products were again analysed after the rust in the pipes and tanks had been washed away, so that the juice dissolved less iron compounds than in the first few days.

| Class of Sugar. | No. | Mgrms. Iron in 100 grms. | | |
|---|---|---|---|---|
| | | Sugar. | Masse cuite. | Mother Liquor. |
| No. 25 D.S. | I. | 4·6 | 5·5 | |
| | II. | 1·0 | 4·5 | |
| No. 16 D.S. | I. | 1·5 | 2·3 | |
| | II. | 2·3 | 2·3 | |
| No. 17 D.S. | I. | 7·0 | 2·3 | |
| | II. | 1·8 | — | |
| Refining Crystals | I. | 2·3 | 4·6 | |
| | II. | 1·6 | 4·6 | |
| ,, ,, | I. | 1·8 | 4·6 | |
| | II. | 1·0 | 4·6 | |
| ,, ,, | I. | 4·6 | 5·2 | 7·0 |
| | II. | 1·2 | 3·5 | 5·5 |
| ,, ,, | I. | 2·3 | 4·6 | |
| | II. | 1·5 | — | |
| ,, ,, | I. | 5·5 | 4·6 | 7·0 |
| | II. | 1·1 | 1·6 | 2·3 |
| ,, ,, | I. | 2·0 | 4·6 | 4·0 |
| | II. | 1·5 | 4·6 | |
| ,, ,, | I. | 35·— | 25·5 | |
| | II. | 2·3 | 2·3 | |
| ,, ,, | I. | 4·6 | 7·0 | 9·3 |
| | II. | 3·5 | 7·0 | 9·3 |
| ,, ,, | I. | 3·5 | 5·5 | |
| | II. | 1·2 | 3·5 | |
| | | 9·3 | 7·0 | |
| | | 1·6 | 2·3 | |
| Black-Stroop | | | 7·0 | |
| Waste Molasses | | | 31·0 | |
| Black Candy | | | 1·8 | |
| Sucrose coloured with Caramel and Iron | | | 93·— | |
| ,, ,, ,, ,, | | | 16·— | |
| ,, ,, Iron Citrate | | | 64·— | |

The above figures represent milligrammes of iron in 100 grms. of sugar, masse cuites, and mother-liquor in the latter. The figures I. and II. refer to samples taken at the beginning and middle of the grinding season respectively.

Although the percentages of iron are very trifling, we see that the masse cuites and sugars contain more iron at the commencement of each season than afterwards. The highest figure was 35 mgrms. per 100 grms. of sugar, and in this case the colour was deep brown and the taste very metallic. A sugar solution mixed on purpose with iron citrate or with caramel and iron saccharate, after evaporation and crystallization, yielded an almost black sugar, having a very pronounced metallic taste, and containing not less than 100 mgrms. of iron per 100 grms.

Sucrose crystallized from a solution containing iron citrate and caramel gave a polarization of 99, and contained 16 mgrms. of iron, and had a colour corresponding with No. 14 D.S.

The proportions of iron found in the masse cuites and in the sugars obtained from these show very strikingly that the iron has been fixed by the sugar crystals and is not dissolved in the molasses; otherwise, the masse cuites (containing the molasses) would certainly have contained more iron than the sugars which are only covered with a film of molasses, while the table proves the contrary.

A few samples of masse cuites were allowed to drain in funnels lined with copper gauze, when the filtered molasses contained nearly as much iron as the crystallized sugar, this being a direct proof that the iron had been incorporated in the sugar crystals and not merely dissolved in the molasses. Further, the table shows that although traces of iron can cause a dark colour, all coloration of the crystal need not be due to that substance. The black-stroop and black candy do not contain notable quantities of iron, and yet they are very dark, showing that other substances, *e.g.*, caramel, can have the same effect.

**Definition of First and Second Sugars.**

Merchants often require a strict definition of firsts and seconds, and in former years this distinction was easily made. Firsts were obtained directly from syrup, and seconds were obtained by the concentration and curing of molasses. At the present day, molasses boiled to grain yield a product which cannot be distinguished from a finely grained first product; and, on the other hand, a mixture of syrup and molasses boiled smooth produces a product which closely resembles second sugar, so that a distinction is difficult, if not impossible. It has been suggested that the smell is a good characteristic of second sugar, but it is evident that this indication is much too uncertain to decide whether a given parcel of sugar is really firsts or seconds.

**Composition of various Cane Sugars.**

Although the list is by no means complete, the following are analyses of cane sugars produced in various countries:—

| Description. | Polarization. | Reducing Sugar. | Ash. | Moisture. | Undetermined. | Nett. |
|---|---|---|---|---|---|---|
| JAVA. | | | | | | |
| White sugar over No. 25 D.S. | 99·6 | 0·05 | 0·02 | 0·06 | 0·27 | |
| ,, ,, No. 20 D.S. | 99·4 | 0·10 | 0·02 | 0·18 | 0·30 | |
| Cubes .. .. .. .. .. | 99·2 | 0·12 | 0·05 | 0·26 | 0·37 | |
| Pilé sugar .. .. .. .... | 99·3 | 0·10 | 0·02 | 0·16 | 0·42 | |
| Raw sugar No. 18 D.S. . | 98·8 | 0·30 | 0·13 | 0·31 | 0·46 | 97·85 |
| ,, No. 17 D.S.... | 98·6 | 0·40 | 0·22 | 0·40 | 0·38 | 97·10 |
| Refining crystals .. .. .. | 97·5 | 0·60 | 0·35 | 0·66 | 0·89 | 95·15 |
| ,, ,, .... ... | 97·0 | 0·76 | 0·23 | 1·00 | 1·01 | 95·09 |
| ,, ,, .. .. . | 96·5 | 0·84 | 0·47 | 1·12 | 1·07 | 93·41 |
| White seconds, No. 25 D.S. | 99·0 | 0·20 | 0·13 | 0·20 | 0·47 | 98·15 |
| Second sugar, No. 18 D.S.. | 97·2 | 1·01 | 0·38 | 0·82 | 0·59 | 94·29 |
| ,, ,, No. 16 D.S.. | 97·0 | 0·85 | 1·10 | 0·53 | 0·52 | 90·65 |
| ,, ,, No. 14 D.S.. | 95·5 | 1·40 | 0·95 | 1·24 | 0·99 | 89·35 |
| ,, ,, No. 12 D.S.. | 94·0 | 1·78 | 1·11 | 1·60 | 1·51 | 86·67 |
| Molasses sugar .. .. ... | 82·4 | 5·81 | 2·27 | 5·37 | 4·15 | 65·24 |
| Black-stroop .. .. .. .. | 71·59 | 7·14 | 3·81 | 6·30 | 11·16 | 45·41 |
| Concrete sugar .. .. .... | 75·5 | 9·60 | 2·50 | 5·60 | 6·80 | 53·40 |
| ,, ,, .... .. .. | 84·8 | 2·74 | 2·25 | 5·24 | 4·97 | 74·81 |
| PHILIPPINES. | | | | | | |
| Mat sugar .. .. .. .. | 90·1 | 3·51 | 1·08 | 1·38 | 3·93 | 81·19 |
| ,, ,, .. .. .... | 84·0 | 7·02 | 1·62 | 2·04 | 5·32 | 68·88 |
| ,, ,, .. .. .. .. | 80·5 | 9·56 | 3·06 | 2·77 | 4·11 | 55·64 |
| HAWAII. | | | | | | |
| Refining crystals .. .. .. | 96·61 | 0·83 | 0·54 | 1·12 | 0·91 | 93·07 |
| ,, ,, .... .... | 96·0 | 1·32 | 0·77 | 0·96 | 0·95 | 90·83 |
| ,, ,, .. .. .. | 95·4 | 0·77 | 0·74 | 1·06 | 2·03 | 90·93 |
| LOUISIANA. | | | | | | |
| Refining crystals .. .. . | 96·1 | 1·47 | 0·50 | 0·96 | 0·97 | 92·13 |
| CUBA. | | | | | | |
| 1st sugar . .. .. .... | 96·4 | 0·67 | 0·54 | 6·54 | 0·85 | 93·03 |
| Molasses sugar .. .. .. | 77·4 | 9·61 | 1·55 | 5·17 | 6·27 | 60·04 |
| PORTO RICO. | | | | | | |
| Refining crystals .. .. .. | 95·8 | 1·40 | 0·43 | 1·06 | 1·31 | 92·25 |
| EGYPT. | | | | | | |
| 1st sugar .. .. .... | 97·9 | 0·50 | 0·31 | 0·58 | 0·71 | 95·85 |
| 2nd ,, .... .. .. .. | 80·4 | 3·40 | 5·52 | 4·35 | 6·33 | 49·40 |

| Description. | Polarization. | Reducing Sugar. | Ash. | Moisture. | Undetermined. | Nett. |
|---|---|---|---|---|---|---|
| DEMERARA. | | | | | | |
| Yellow crystals (grocery) .. | 95·8 | 1·42 | 0·38 | 1·46 | 0·49 | 92·48 |
| Refining crystals.. .. .. | 95·0 | 1·40 | 0·59 | 1·66 | 1·35 | 90·65 |
| Syrup sugar (grocery) .. | 88·5 | 3·06 | 2·54 | 3·12 | 2·78 | 72·74 |
| ,, ,, ,, .. .. | 82·9 | 3·84 | 3·58 | 6·44 | 3·24 | 61·26 |
| TRINIDAD. | | | | | | |
| Centrifugalled (refining) .. | 97·5 | 0·67 | 0·45 | 0·92 | 0·46 | 94·08 |
| Crystals (grocery) .. .... | 96·5 | 0·99 | 0·38 | 1·26 | 0·87 | 93·61 |
| Syrup sugar (grocery).. .. | 87·7 | 4·39 | 0·68 | 3·66 | 3·56 | 79·91 |
| BARBADOS. | | | | | | |
| Muscovado, grocery .. .. | 88·6 | 5·30 | 0·47 | 3·42 | 2·21 | 80·95 |
| ,, ,, .... | 86·2 | 4·40 | 2·66 | 3·72 | 3·02 | 68·50 |
| ,, ,, .. .. | 83·0 | 5·92 | 2·12 | 4·66 | 4·30 | 66·48 |
| SURINAM. | | | | | | |
| 1st sugar .. .. .. .... | 96·8 | 1·28 | 0·23 | 0·76 | 0·93 | 93·37 |
| 2nd ,, .... .. .. .. | 85·6 | 5·12 | 4·02 | 3·33 | 1·93 | 60·38 |
| GUATEMALA. | | | | | | |
| Syrup sugar (grocery) ... | 86·4 | 7·32 | 0·50 | 3·56 | 2·22 | 76·58 |
| JAMAICA. | | | | | | |
| Syrup sugar (grocery).. .. | 90·3 | 3·08 | 0·97 | 3·74 | 6·91 | 82·33 |
| PERU. | | | | | | |
| Refining crystals .. .... | 97·6 | 0·90 | 0·47 | 0·32 | 0·71 | 94·35 |
| ,, ,, .... | 95·3 | 1·87 | 0·32 | 1·64 | 0·82 | 91·83 |
| Syrup sugar .. .. .... | 84·5 | 1·76 | 4·70 | 4·50 | 4·54 | 59·24 |
| MEXICO. | | | | | | |
| Refining crystals .. .... | 94·7 | 1·58 | 0·63 | 1·98 | 1·11 | 89·97 |
| ,, syrups .. .. .. | 92·6 | 1·47 | 1·19 | 2·60 | 2·14 | 85·18 |
| SAN DOMINGO. | | | | | | |
| Refining syrups .. .... | 85·2 | 4·10 | 1·91 | 4·80 | 3·99 | 71·55 |
| BRAZIL. | | | | | | |
| Refining crystals .. .... | 96·3 | 1·67 | 0·18 | 1·26 | 0·59 | 93·73 |
| Pernambuco molasses.. .. | 80·5 | 6·83 | 1·85 | 5·71 | 5·11 | 63·43 |
| Muscovado .. .. .. .. | 83·9 | 6·98 | 1·02 | 3·84 | 4·26 | 71·82 |

# IV.—Preservation of the Sugar during Storage and Transport.

As sugar must be of good quality, not only when shipped but also when it reaches the consignee, care must be taken to prevent deterioration before delivery.

**Decrease in Polarization.**

As regards decrease in polarization, this may be either very rapid and limited, or slow but continuous.

In Java a difference in polarization has many a time been noticed between the date when the sugar left the factory and its arrival at port a couple of days later. The difference sometimes amounted to about one per cent. and did not subsequently increase, but even decreased in many cases.

The buyers insisted upon a very dry sugar, and stipulated that sugar polarizing under 96·5° was liable to heavy penalties. Each parcel of sugar was analysed before packing (as it left the dryer), in order to be quite sure that no single basket of sugar polarized below the limit of 96·5°. Notwithstanding these precautions, the samples taken from the baskets on their arrival at the shipping port repeatedly showed a lower polarization than those taken at the factory, and consequently the manufacturer sometimes had to pay the penalty. These differences gave rise to complaints from the owners of the factories to their managers, and from these latter against the sugar merchant and his clerks that they had not paid sufficient attention to the sampling, but had left this work to ignorant coolies, &c. As neither side could adduce proofs, the dispute frequently ended in suspicion, or even in dismissal.

In one instance, the polarization at the shipping port was 0·8 per cent. lower than when the same sample was tested at the factory three days previously, and the same difference occurred throughout the whole grinding season. But, on arrival in America, the sugar showed the same polarization as at the factory, proving that there had been no inversion or other decomposition of the sucrose.

But, until this proof was received in Java, it was very naturally concluded that the sugar had deteriorated after leaving the factory, but whether due to inversion, absorption of moisture, or actual decomposition, it was impossible to say, because only the polarization had been determined at the two places. As all these sugars contained rather high percentages of reducing sugars it seemed not

improbable that some glucose might have crystallized out along with the sucrose, giving a birotation which would falsify the direct polarization. This explanation was disproved by the similarity in the ratio of ash to reducing sugars as determined in the finished sugar and in the molasses separated from it: also by obtaining the same polarization in hot and cold solutions, whereby any possible birotation is eliminated.

As a detailed analysis of these sugars was not made immediately after drying, their subsequent examination was not carried out, but the fact that the original polarization decreased after only three days, that such decrease did not continue after that period, but, on the contrary, disappeared entirely, coupled with the fact that these sugars contained much reducing sugar, led to the following hypothesis:—

The crystals under consideration are coated with a layer of artificially dried molasses containing much reducing sugar, and are, consequently, very hygroscopic. The sugar is packed on an estate situated in a dry district of the island, and its original polarization therefore represents well-dried sugar. This is then forwarded to the shipping port where the atmosphere is generally much more moist than at the inland factory. The sample of sugar taken from the bags at once absorbs moisture and shows a lower polarization than at the factory, while the great bulk of the sugar (protected by the bags) retains its original polarization. This sugar ultimately arrives in a country having a more temperate climate and a drier atmosphere, and when another sample is taken from the bags this does not absorb sufficient moisture to influence the polarization, which now strictly represents the bulk, and consequently agrees with the original polarization. The sugar in bulk does not change, but the sample taken for testing absorbs more moisture in a damp climate than in a dry one, and thus causes the differences observed.

**Decrease in Polarization caused by Inversion and Fermentation.**

A much more serious deterioration during storage and transport is due to absorption of moisture and inversion or fermentation.

It is evident that neither inversion nor fermentation are possible unless the sugar be moist, but whether the sugar was originally moist, or has become so by absorption of moisture from the atmosphere is a matter of indifference, as is seen in the following instances.

In former years, refining crystals were sold in Java on the Dutch Standard basis and not on polariscope test, no deduction being made for moisture. As this water was paid for at the same price as sugar, there was much temptation to deliver it as moist as possible, so that deterioration of sugar after delivery became very serious, as a few instances recorded here will show.

| No. | Number of Baskets. | Date of Delivery. | Date of Analysis. | Polarization at the Date of Delivery. | Polarization at the Date of Analysis. | Total Decline. | Decline per Month. |
|---|---|---|---|---|---|---|---|
| | | Refining Crystals. | | | | | |
| 1 | 1150 | Second half of August .. | End of January .. .... | 95·9 | 95·2 | 0·7 | 0·132 |
| 2 | 244 | Beginning of November.. | ,, ,, .. .. | 95·9 | 95·1 | 0·8 | 0·264 |
| 3 | 368 | ,, ,, December.. | Beginning of January.... | 96·6 | 96·1 | 0·5 | 0·457 |
| 4 | 647 | ,, ,, July .. .. | December .. .. .. .. | 95·6 | 94·6 | 1·0 | 0·202 |
| 5 | 1368 | June-July .. .. .... | End of December.. .. | 96·0 | 95·0 | 1·0 | 0.167 |
| 6 | 116 | End of June .. .. .. | ,, September .. . | 97·0 | 95·0 | 2·0 | 0·671 |
| 7 | 309 | Beginning of July .... | ,, August .. .... | 97·6 | 96·8 | 0·8 | 0·392 |
| 8 | 264 | ,, ,, .. .. | ,, ,, .. .. .. | 98·0 | 96·6 | 1·4 | 0·694 |
| 9 | 590 | Middle June .. .. .... | Beginning of November. | 96·7 | 96·3 | 0·4 | 0·086 |
| 10 | 470 | June .. .. .. .. .. | ,, ,, .. | 97·0 | 96·6 | 0·4 | 0·063 |
| 11 | 1266 | Beginning of August .... | February .. .. .. .. | 98·3 | 97·6 | 0·7 | 0·120 |
| 12 | 905 | End of May .. .. .. | September .. .. .... | 96·5 | 96·0 | 0·5 | 0·125 |
| 13 | 157 | ,, ,, .. .. .... | End of November .. .. | 97·2 | 91·8 | 5·4 | 0·896 |
| 14 | 490 | Middle September .. .. | November .. .. .... | 97·2 | 96·9 | 0·3 | 0·123 |
| | | Sugar, Nos. 16-18 D.S. | | | | | |
| 15 | 974 | 15th September.. .. .. | 15th October .. .. .. | 98·0 | 96·9 | 1·1 | 0·37 |
| 16 | 714 | 25th August .. .. .... | February .. .. .... | 98·8 | 96·4 | 2·4 | 0·40 |
| 17 | 679 | 1st September .. .. .. | Middle February .. . | 98·3 | 97·3 | 1·0 | 0·21 |
| 18 | 2556 | September .. .. .... | ,, ,, .. .... | 98·6 | 97·4 | 1·2 | 0·28 |

A lot of Hongkong refined sugar packed in bags and lying in a moist place lost about five per cent. in polarization during four months; and then contained 2·25 per cent. of water, and 2·35 of reducing sugar. As the sugar originally contained only a trace of reducing sugar and of moisture, it is obvious that the deterioration is not due to salts, reducing sugars, or acids, as is sometimes pretended, but solely to fermentation and inversion, after absorption of moisture.

As soon as it was recognised that moisture is the chief or primary factor in deterioration, the Java merchants enforced a heavy penalty on sugar polarizing under 96·5°, but paid more for every per cent. over 96·5° than at that figure.* It thus became desirable for the manufacturers to dry their sugar as much as possible, first, in order to avoid delivering under 96·5°, and secondly to allow more sugar to polarize over 96·5 than under that figure, so that after this alteration in the selling conditions, the Java sugar was sold dry.

* The price quoted for refining crystals is on a basis of 96·5 polarization. Sugar polarizing above that figure is paid 9 cents per picul for each additional per cent. polarization, while the price for 96·5° sugar is always under 9 guilders per picul, and ranges from 6 to 7. At a price of 6 guilders a picul, refining sugar of 96·5° is therefore sold for 6 guilders, and one of 97·5° is sold at f. 6·09.

One fortunate result was that complaints and claims for deterioration during storing and transport became fewer, although, in some cases, even well-dried sugar absorbed water, and consequently deteriorated. The satisfactory keeping qualities of Java sugars are illustrated in the table given below, representing several hundred thousand tons of sugar shipped to America.

The polarization which served as a basis for payment in Java is the average of the two polarizations by the analysts employed by the manufacturer and exporter. In the same way, the figure for polarization in America is the average of the two polarizations by the analysts employed by the importer and the refiner. These figures are therefore as accurate as possible, although, of course, small errors are not excluded. When considering the figures, we must remember that the polarization has been made at a higher temperature in Java than in America, which causes the former to be somewhat low. As the difference is about 0·2 degrees Ventzke, sugar polarizes 0·2 per cent. higher in America than in Java, so that a sugar of 97·0 in Java, will polarize 97·2 in America.

With the exception of a few large differences, and others due to analytical errors, it will be seen that, on arrival, the sugars usually polarize less than the original figure by 0·2. The differences which are not very considerable are due to absorption of moisture during the long sea voyage, and deterioration, properly speaking; so that the drying of these sugars has greatly improved their keeping qualities.

Before moisture was recognised as the principal factor in deterioration, the rapid deterioration of certain sugars was ascribed to the size of the grain, the method of clarification, or to the fact that molasses had been returned in the first masse cuites. But the table (page 287) shows that none of these circumstances influence the keeping power of the sugars.

With a view to investigating the rapidity of deterioration of packed sugar when stored in warehouses during the rainy season, baskets of sugar from different factories were put aside, and every month a sample was taken in the usual manner and analysed. After 12 months, the contents of the baskets were mixed and the mixed sugar from every basket likewise analysed. The monthly figures of a few baskets taken at random and the initial and final analyses follow here.

The sugars 1-23 were packed in baskets, and No. 24 in a bag.

In most cases, the sugar remained unchanged during the first few months, then attracted moisture and afterwards became partially inverted. The increase in undetermined matter is only apparent, since the levo-rotation of the invert sugar neutralizes a part of the dextro-rotation of the sucrose, thus causing it to appear less than it really is and proportionally increasing the balance of undetermined matters, which are found by difference.

| No. | Method of Clarification. | Size of the Crystals. | Are Molasses returned? | Shipped in | Polarization in Java. | Polarization in America. | Difference. |
|---|---|---|---|---|---|---|---|
| 1 | Defecation. | Middling. | Yes. | June. | 97·72 | 95·95 | −1·77 |
| 2 | ,, | ,, | ,, | July | 97·55 | 95·8 | −1·75 |
| 3 | ,, | Fine. | No. | June. | 98·45 | 97·05 | −1·40 |
| 4 | ,, | Small. | ,, | September. | 97·4 | 96·4 | −1·00 |
| 5 | ,, | Fine. | Yes. | July. | 97·78 | 97·1 | −0·68 |
| 6 | ,, | Small. | ,, | September. | 97·6 | 97·1 | −0·6 |
| 7 | ,, | Irregular. | ,, | ,, | 97·2 | 96·6 | −0·6 |
| 8 | ,, | Small. | ,, | ,, | 97·8 | 97·2 | −0·6 |
| 9 | ,, | Coarse. | ,, | July. | 97·92 | 97·45 | −0·47 |
| 10 | ,, | Middling. | ,, | June. | 98·12 | 97·8 | −0·32 |
| 11 | ,, | ,, | ,, | ,, | 97·5 | 97·2 | −0·30 |
| 12 | ,, | ,, | ,, | July. | 96·86 | 96·6 | −0·26 |
| 13 | ,, | Fine. | ,, | June. | 97·9 | 97·65 | −0·25 |
| 14 | Carbonatation. | Middling. | No. | ,, | 97·12 | 96·9 | −0·22 |
| 15 | Defecation. | Small. | Yes. | July. | 97·85 | 97·65 | −0·20 |
| 16 | ,, | Irregular. | ,, | ,, | 97·35 | 97·25 | −0·10 |
| 17 | ,, | Fine. | ,, | June. | 98·05 | 97·95 | −0·10 |
| 18 | ,, | ,, | ,, | August. | 97·99 | 97·9 | −0·09 |
| 19 | ,, | ,, | ,, | ,, | 98·36 | 98·30 | −0·06 |
| 20 | ,, | ,, | No. | ,, | 97·77 | 97·72 | −0·05 |
| 21 | ,, | Middling. | Yes. | July. | 97·79 | 97·8 | +0·01 |
| 22 | ,, | Coarse. | ,, | ,, | 97·22 | 97·25 | +0·03 |
| 23 | ,, | ,, | ,, | October. | 97·47 | 97·5 | +0·03 |
| 24 | Carbonatation. | Fine. | ,, | September. | 96·37 | 96·4 | +0·03 |
| 25 | Defecation. | ,, | ,, | August. | 98·02 | 98·1 | +0·08 |
| 26 | ,, | ,, | ,, | ,, | 98·08 | 98·16 | +0·08 |
| 27 | Carbonatation. | ,, | No. | ,, | 97·43 | 97·55 | +0·12 |
| 28 | ,, | Middling. | Yes. | September. | 97·07 | 97·25 | +0·18 |
| 29 | Defecation. | Fine. | ,, | August. | 97·76 | 97·95 | +0·19 |
| 30 | Carbonatation. | ,, | ,, | July. | 96·68 | 96·90 | +0·22 |
| 31 | Defecation. | Coarse. | No. | June. | 97·07 | 97·35 | +0·28 |
| 32 | ,, | Middling. | Yes. | October. | 97·09 | 97·38 | +0·29 |
| 33 | ,, | Irregular. | No. | July. | 97·97 | 98·30 | +0·33 |
| 34 | Carbonatation. | Fine. | Yes. | August. | 97·06 | 97·49 | +0·43 |
| 35 | Defecation. | ,, | ,, | October. | 97·66 | 98·10 | +0·44 |

**The Decrease in Quality is not so great in Practice.**

The decrease in polarization shown in the tables is much higher than in practical working. In these experiments, every basket was exposed to the atmosphere on all sides, whereas, in practice, they are heaped up in piles and only a few baskets have one side exposed to the moist air. Further, the experiment did not represent the circumstances met with in practice, because every basket was pierced monthly

when sampled: the packing material was thereby severely damaged and many germs could have entered through the numerous holes. Owing to the repeated sampling the contents of the basket became looser in some places, and the deterioration must have gone further, than in the parts still remaining untouched. When the sample was drawn, first from a loosened part and, a month later, from a fresh area, it may have occurred that the later analysis yielded much better figures than the former, making it appear as though the quality of the sugar had first deteriorated and then improved.

| Analysis. | At the start. | After 1 Month. | After 2 Months. | After 3 Months. | After 4 Months. | After 5 Months. | After 6 Months. | After 7 Months. | After 8 Months. | After 9 Months. | After 10 Months. | After 11 Months | Mixed Contents after 12 Months |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Polarization .. .. .. | 99·00 | 98·90 | 98·90 | 98·70 | 98·70 | 98·20 | 98·40 | 97·00 | 97·10 | 97·00 | 96·30 | 95·90 | 96·50 |
| Reducing sugar .. .... | 0·40 | 0·41 | 0·41 | 0·51 | 0·50 | 0·85 | 0·75 | 1·37 | 1·12 | 1·15 | 1·53 | 1·83 | 1·53 |
| Ash .. .. .. .. .. | 0·16 | 0·16 | 0·16 | 0·17 | 0·16 | 0·16 | 0·16 | 0·16 | 0·16 | 0·17 | 0·16 | 0·16 | 0·18 |
| Water .. .. .. .... | 0·13 | 0·15 | 0·14 | 0·26 | 0·26 | 0·49 | 0·34 | 0·94 | 1·02 | 1·00 | 1·20 | 1·20 | 0·92 |
| Undetermined .. .. | 0·31 | 0·38 | 0·39 | 0·36 | 0·38 | 0·30 | 0·35 | 0·53 | 0·60 | 0·68 | 0·81 | 0·91 | 0·87 |
| Total .. .. .... | 100·— | 100·— | 100·— | 100·— | 100·— | 100·— | 100·— | 100·— | 100·— | 100·— | 100·— | 100·— | 100·— |
| Nett .. .. .. .. .. | 97·80 | 97·69 | 97·69 | 97·34 | 97·40 | 96·55 | 96·85 | 94·83 | 95·18 | 95·00 | 93·99 | 93·27 | 94·07 |
| Polarization .. .. .. | 98·30 | 98·30 | 98·30 | 97·90 | 97·70 | 96·90 | 96·10 | 96·40 | 94·80 | 94·80 | 94·20 | 96·60 | 95·00 |
| Reducing sugar .. .... | 0·59 | 0·56 | 0·56 | 0·76 | 0·87 | 1·23 | 1·74 | 1·43 | 2·25 | 2·13 | 2·64 | 1·36 | 2·39 |
| Ash .. .. .. .. .. | 0·27 | 0·28 | 0·28 | 0·28 | 0·27 | 0·28 | 0·27 | 0·28 | 0·27 | 0·27 | 0·27 | 0·29 | 0·27 |
| Water .. .. .. .... | 0·50 | 0·56 | 0·46 | 0·46 | 0·56 | 0·80 | 1·13 | 1·00 | 1·64 | 1·54 | 1·62 | 1·02 | 1·12 |
| Undetermined .. .. | 0·34 | 0·32 | 0·44 | 0·60 | 0·60 | 0·79 | 0·76 | 0·89 | 1·04 | 1·26 | 1·27 | 0·73 | 1·22 |
| Total .. .. .... | 100·— | 100·— | 100·— | 100·— | 100·— | 100·— | 100·— | 100·— | 100·— | 100·— | 100·— | 100·— | 100·— |
| Nett .. .. .. .. .. | 96·36 | 96·36 | 96·36 | 95·74 | 95·48 | 94·27 | 93·01 | 93·57 | 91·20 | 91·32 | 90·21 | 93·79 | 91·26 |
| Polarization .. .. .. | 97 00 | 97·30 | 97·20 | 97·00 | 97·00 | 97·00 | 96·70 | 96·10 | 96·40 | 96·50 | 96·20 | 95·50 | — |
| Reducing sugar .. .... | 1·19 | 1·07 | 1·18 | 1·19 | 1·16 | 1·16 | 1·01 | 1·18 | 1·08 | 1·53 | 1·74 | 1·83 | — |
| Ash .. .. .. .. .. | 0·32 | 0·31 | 0·31 | 0·32 | 0·31 | 0·31 | 0·31 | 0·30 | 0·30 | 0·30 | 0·32 | 0·29 | — |
| Water .. .. .. .... | 0·60 | 0·54 | 0·67 | 0·78 | 0·76 | 0·80 | 1·30 | 1·50 | 1·53 | 0·98 | 0·94 | 1·22 | — |
| Undetermined .. .. | 0·89 | 0·78 | 0·64 | 0·71 | 0·77 | 0·73 | 0·68 | 0·92 | 0·69 | 0·69 | 0·80 | 1·16 | — |
| Total .. .. .... | 100·— | 100·— | 100·— | 100·— | 100·— | 100·— | 100·— | 100·— | 100·— | 100·— | 100·— | 100·— | — |
| Nett .. .. .. .. .. | 94·21 | 94·68 | 94·47 | 94·21 | 94·29 | 94·29 | 94·04 | 93·42 | 93·82 | 93·47 | 92·86 | 92·22 | — |
| Polarization .. .. .. | 96·60 | 97·00 | 97·00 | 97·20 | — | 97·50 | 97·30 | 96·50 | 95·65 | 95·80 | 96·60 | 96·70 | 96·70 |
| Reducing sugar .. .... | 1·12 | 1·12 | 1·16 | 1·16 | — | 0·52 | 0·50 | 0·87 | 1·42 | 1·60 | 1·53 | 1·30 | 1·28 |
| Ash .. .. .. .. .. | 0·40 | 0·39 | 0·39 | 0·40 | — | 0·40 | 0·40 | 0·39 | 0·39 | 0·39 | 0·40 | 0·38 | 0·39 |
| Water .. .. .. .... | 1·10 | 0·94 | 0·82 | 0·60 | — | 0·98 | 1·10 | 1·56 | 1·26 | 1·14 | 0·76 | 1·00 | 0·86 |
| Undetermined .. .. | 0·78 | 0·55 | 0·63 | 0·64 | — | 0·60 | 0·70 | 0·68 | 1·28 | 1·07 | 0·71 | 0·62 | 0·77 |
| Total .. .. .... | 100·— | 100·— | 100·— | 100·— | — | 100·— | 100·— | 100·— | 100·— | 100·— | 100·— | 100·— | 100·— |
| Nett .. .. .. .. .. | 93·48 | 93·93 | 93·89 | 94·04 | — | 94·98 | 94·80 | 93·68 | 92·28 | 92·25 | 93·07 | 93·50 | 93·47 |

| Analysis at the | I. | | II. | | III. | | IV. | | V. | | VI. | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Begin of the Experiment. | End | Begin of the Experiment. | End | Begin of the Experiment. | End | Begin of the Experiment. | End | Begin of the Experiment. | End | Begin of the Experiment. | End |
| Polarization | 97·40 | 97·10 | 97·70 | 94·00 | 98·30 | 93·70 | 98·50 | 92·60 | 99·00 | 98·50 | 97·90 | 96·50 |
| Reducing sugar | 0·93 | 1·28 | 0·92 | 2·47 | 0·66 | 2·74 | 0·49 | 2·39 | 0·40 | 1·53 | 0·53 | 1·50 |
| Ash | 0·34 | 0·33 | 0·29 | 0·31 | 0·28 | 0·25 | 0·21 | 0·21 | 0·16 | 0·18 | 0·38 | 0·34 |
| Water | 0·68 | 0·68 | 0·60 | 1·74 | 0·54 | 2·14 | 0·53 | 3·26 | 0·13 | 0·92 | 0·50 | 0·96 |
| Undetermined | 0·65 | 0·61 | 0·49 | 1·48 | 0·22 | 1·27 | 0·17 | 1·54 | 0·31 | 0·87 | 0·69 | 0·70 |
| Total | 100·— | 100·— | 100·— | 100·— | 100·— | 100·— | 100·— | 100·— | 100·— | 100·— | 100·— | 100·— |
| Nett | 94·77 | 94·17 | 95·33 | 89·98 | 96·24 | 90·71 | 96·96 | 88·16 | 97·80 | 94·07 | 95·47 | 93·30 |

| | VII. | | VIII. | | IX. | | X. | | XI. | | XII. | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Polarization | 98·00 | 92·70 | 98·30 | 95·00 | 97·50 | 95·60 | 97·50 | 96·00 | 97·10 | 93·60 | 96·60 | 96·70 |
| Reducing sugar | 0·87 | 2·56 | 0·59 | 2·39 | 1·01 | 1·74 | 1·01 | 1·60 | 1·12 | 2·56 | 1·12 | 1·28 |
| Ash | 0·26 | 0·25 | 0·27 | 0·27 | 0·30 | 0·32 | 0·37 | 0·36 | 0·55 | 0·47 | 0·40 | 0·39 |
| Water | 0·29 | 2·46 | 0·50 | 1·12 | 0·56 | 1·36 | 0·59 | 1·22 | 0·59 | 1·94 | 1·10 | 0·86 |
| Undetermined | 0·58 | 2·03 | 0·34 | 1·22 | 0·63 | 0·98 | 0·53 | 0·82 | 0·64 | 1·43 | 0·78 | 0·77 |
| Total | 100·— | 100·— | 100·— | 100·— | 100·— | 100·— | 100·— | 100·— | 100·— | 100·— | 100·— | 100·— |
| Nett | 95·83 | 88·89 | 96·36 | 91·26 | 94·99 | 92·26 | 94·64 | 92·60 | 93·23 | 89·09 | 93·48 | 93·47 |

| | XIII. | | XIV. | | XV. | | XVI. | | XVII. | | XVIII. | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Polarization | 97·20 | 94·40 | 97 30 | 95·40 | 97·50 | 94·70 | 97·80 | 95·40 | 96·80 | 94·10 | 97·20 | 94·80 |
| Reducing sugar | 1·06 | 2·02 | 1·03 | 2·40 | 0·81 | 2·02 | 0·91 | 2·25 | 1·32 | 2·39 | 1·06 | 1·92 |
| Ash | 0·39 | 0·38 | 0·39 | 0·34 | 0·38 | 0·35 | 0·19 | 0·20 | 0·45 | 0·43 | 0·47 | 0·45 |
| Water | 0·75 | 1·96 | 0·56 | 0·86 | 0·52 | 1·74 | 0·38 | 1·34 | 0·66 | 2·06 | 0·68 | 1·26 |
| Undetermined | 0·60 | 1·24 | 0·72 | 1·00 | 0·79 | 1·19 | 0·72 | 0·81 | 0·77 | 1·02 | 0·59 | 1·57 |
| Total | 100·— | 100·— | 100·— | 100·— | 100·— | 100·— | 100·— | 100·— | 100·— | 100·— | 100·— | 100·— |
| Nett | 94·19 | 90·48 | 94·32 | 91·30 | 94·79 | 90·93 | 95·94 | 92·15 | 93·23 | 91·52 | 93·79 | 90·63 |

| | XIX. | | XX. | | XXI. | | XXII. | | XXIII. | | XXIV. | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Polarization | 97·00 | 96·70 | 97·30 | 94·30 | 97·00 | 95·20 | 97·00 | 95·50 | 97·90 | 96·00 | 97·30 | 91·50 |
| Reducing sugar | 1·23 | 1·30 | 0·92 | 2·84 | 1·19 | 2·13 | 1·19 | 1·83 | 0·96 | 2·19 | 1·10 | 3·49 |
| Ash | 0·29 | 0·28 | 0·30 | 0·29 | 0·33 | 0·31 | 0·32 | 0·29 | 0·23 | 0·24 | 0·26 | 0·31 |
| Water | 0·70 | 0·97 | 0 56 | 1·80 | 0·66 | 1·28 | 0·60 | 1·22 | 0·44 | 0·78 | 0·64 | 2·68 |
| Undetermined | 0·78 | 0·75 | 0·92 | 0·77 | 0·82 | 1·08 | 0·89 | 1·16 | 0 47 | 0·79 | 0·70 | 2·02 |
| Total | 100·— | 100·— | 100·— | 100·— | 100·— | 100·— | 100·— | 100·— | 100·— | 100·— | 100·— | 100·— |
| Nett | 94·32 | 94·00 | 94·88 | 90·01 | 94·16 | 91·52 | 94·21 | 92·22 | 95·79 | 92·61 | 94·90 | 86·46 |

But notwithstanding the fact that this experiment failed as a quantitative measure of the deterioration, it afforded a clue as to the cause, namely the absorption of water, followed by a simple inversion by fungi and yeasts. Water alone cannot invert at the temperatures which prevail in Java, otherwise sterilized sucrose solutions would be rapidly inverted, which they are not. Neither can fungi attack dry sugar, but when both causes

**Causes of Deterioration.**

co-operate, *i.e.*, when fungi or yeasts can contaminate moist sugar, inversion proceeds very rapidly; the new-formed inverted sugar attracts more moisture with the result that a syrupy solution of inverted sugar oozes out from the baskets and bags.

The analysis of such a syrupy liquid gave these figures:—

| | |
|---|---|
| Polarization | 9·80 |
| Reducing Sugar | 48·00 |
| Ash | 0·95 |
| Brix | 75·4 |

from which the following composition may be calculated:—

| | Per cent. |
|---|---|
| Sucrose | 25 |
| Reducing sugar | 48 |
| Ash | 1 |
| Water | 25 |
| Undetermined | 1 |

This shows much inversion: the sugar itself had also decreased in polarization.

**Action of Micro-organisms.**

The best proof that deterioration is brought about by the simultaneous presence of water and micro-organisms is furnished by an experiment in which portions of dry and of moist sugar (some of which were sterilized and others not), with pieces of the packing material, were preserved in tubes during a couple of months. Every month one tube out of each set was opened and the contents analysed. With a view to ascertaining whether an alkaline reaction of the sugar might prevent inversion, one sample of sterile and unsterile sugar was moistened by a two per cent. sodium carbonate solution.

| | At the start. | | | | After 1 month. | | | | After 4 months. | | | | After 6 months. | | | | After 8 months. | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Polarization | Red. Sugar. | Water. | Reaction. | Polarization | Red. Sugar. | Water. | Reaction. | Polarization | Red. Sugar. | Water. | Reaction. | Polarization | Red. Sugar. | Water. | Reaction. | Polarization | Red. Sugar. | Water. | Reaction. |
| *terilized.* | | | | | | | | | | | | | | | | | | | | |
| )ry.. .. .. | 99·6 | trace | 0·09 | neutr. | 99·6 | trace | 0·05 | neutr. | 99·6 | trace | 0·16 | neutr. | 99·6 | trace | 0·06 | neutr. | 99·5 | trace | 0·10 | neutr. |
| + 1% water.. | 98·8 | ,, | 0·95 | ,, | 98·7 | 0·10 | 0·90 | ,, | 99·2 | 0·32 | 0·44 | ,, | 98·6 | 0·48 | 0·57 | ,, | 98·7 | 0·70 | 0·48 | acid. |
| + 2% ,, | 97·9 | ,, | 1·90 | ,, | 97·6 | 0·10 | 1·80 | ,, | 97·1 | 0·96 | 0·94 | ,, | 96·5 | 1·28 | 0·90 | ,, | 96·0 | 1·44 | 0·95 | ,, |
| + 2% soda .. | 98·1 | ,, | 1·75 | alk. | 98·0 | 0·15 | 1·63 | alk. | 97·1 | 0·53 | 1·90 | alk. | Glass broken. | | | | | | | |
| *Not sterilized* | | | | | | | | | | | | | | | | | | | | |
| )ry.. .. .. | 99·6 | ,, | 0·09 | neutr. | 99·6 | trace | 0·18 | neutr. | 99·2 | 0·28 | 0·16 | neutr. | 98·5 | 0·71 | 0·30 | acid. | 97·7 | 1·40 | 0·60 | ,, |
| + 1% water.. | 98·7 | ,, | 1·01 | ,, | 97·5 | 0·92 | 0·58 | ,, | 96·8 | 1·07 | 1·15 | ,, | 96·1 | 1·92 | 0·82 | ,, | 95·8 | 1·90 | 0·91 | ,, |
| + 2% ,, | 98·0 | ,, | 1·83 | ,, | 95·5 | 1·72 | 2·10 | ,, | 93·7 | 2·32 | 2·20 | ,, | 93·8 | 3·19 | 2·25 | ,, | 90·5 | 3·70 | 1·85 | ,, |
| + 2% soda .. | 97·7 | ,, | 2·11 | alk. | 97·1 | 0·53 | 2·24 | alk. | 94·0 | 2·13 | 2·03 | ,, | 92·3 | 3·33 | 2·02 | neutr | 90·7 | 3·88 | 2·11 | neutr. |

This experiment proves that dry sugar, even when contaminated with germs, remains unchanged, but unsterilized sugar in a moist condition is at once attacked and partly inverted by the germs, though the original reaction be neutral. Sugar in which the micro-organisms have been killed as far as is possible (sugar cannot be completely sterilized at high temperature without becoming decomposed) also deteriorates, but by no means as quickly as when unsterilized. Alkalinity of the sugar is not a sufficient protection against inversion. So long as the reaction is still strongly alkaline (0·2 per cent. of sodium carbonate) inversion proceeds slowly, but as soon as this is neutralized (either by acids formed through the action of the fungi or from the glucose), there is no difference between neutral and alkaline sugars in this respect. Kamerling* showed that sugar coming out of the dryer is practically sterile, and that the source of infection is to be sought in the manipulations during packing and in the packing material itself. Indeed, numerous germs cling to the bags in which the sugar is packed, including those organisms which feed on sugar and invert it.

This latter fact has been observed as far back as 1874 by Serrurier, who ascribed the formation of a moist layer of inverted sugar against the sides of the baskets arriving in Amsterdam, to the influence of the mats in which the sugar was packed. This portion of the sugar contained 7–10 per cent. of reducing sugar, while that in the centre contained not more than one per cent. The fact that the mixed sugar contained only 1½ per cent. of reducing sugar proves that the amount of damaged sugar was inconsiderable and that the inversion had, therefore, confined itself to the outer layers.

**Micro-organisms which cause Deterioration.**

Among the micro-organisms which cause deterioration, Maxwell† mentions the organisms of the butyric and lactic acid fermentations, while Storey ascribes it to fungi, especially the *Penicillium glaucum*. Kamerling‡ found in dried sugar chiefly a fungus flora related to *Penicillium* (over 20 varieties were observed, and not less than 19 belonged to the group *Sterigmatocytes*, *Aspergillus*, *Penicillium* and *Citromyces*.) After the sugar has become very moist, the yeasts predominate, and among these are *Saccharomyces*, *Torula*, *Monilia*, &c., which invert sugar strongly, so that during the course of deterioration of raw sugar, the first attack is made by fungi, and after inversion and absorption of moisture have commenced, they are superseded by yeasts which play the principal part in the deterioration.

Greig-Smith and Steel§ ascribed it to a bacillus which they called *Bacillus levaniformans*, and Noël Deerr and Norris‖ report having found five different species of bacteria, none of which could positively be identified with the *Bacillus levaniformans*.

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* *Archief voor de Java Suikerindustrie*, 1903, 122. † *Louisiana Planter*, 1896, 154.
‡ *Archief voor de Java Suikerindustrie*, 1899, 629.
*Journ. Chem. Ind., International Sugar Journal*, 1903, 448.
‖ *International Sugar Journal*, 1908, 333.

Although different investigators ascribe deterioration to different organisms, they all agree that some organism or other is the direct cause, but that the sugar must be moist to allow of their growth and activity. A dry sugar can be kept unaltered for a long time, while a moist one soon becomes inverted and falls off in quality, owing to the activity of micro-organisms.

**Preventing Deterioration.** There are therefore two methods of preventing deterioration during storage or transport. First, by eliminating the micro-organisms by sterilizing the packing material, and, second, by drying the sugar and taking care that it does not re-absorb moisture, so that even if the spores or germs are present they cannot develop and thus remain inoffensive.

**Disinfected Packing Material.** Kamerling* found that disinfection of the packing material by means of a hot one per cent. solution of carbolic acid is sufficient to kill the germs of the destructive organisms, and suggested treating all the packing material with this disinfectant before bringing it into contact with the sugar.

Wishing to test this point, the author requested the Director of the Netherlands-India Agricultural Society (Nederlandsche Indische Landbouw Maatschappij) to induce the managers of all the factories belonging to that Company to pack one kind of sugar in disinfected and non-disinfected material respectively, to allow one package of every kind to stand over during a year and to send a sample of the mixed contents of every package to the Experimental Station. The mats, baskets, and bags were dipped in a hot one per cent. carbolic acid solution and dried, after which they were used for packing the sugar.

The analyses of the sugars at the commencement and at the end of the experiment are recorded on next page.

During the period of storing, the decrease in polarization was caused by absorption of moisture quite as much as by inversion, consequently the first mentioned factor has been eliminated in the above tables by calculating the polarization and reducing sugar on 100 parts of dry substance, leaving the difference due solely to the action of the inversion. We notice that the sugar in the disinfected packing material was also partially inverted, but to a less extent than in the unsterilized bags, and that this was the case with bags as well as with baskets.

**Impermeable Packing Material.** As it appeared that disinfection is not sufficient to protect the sugar against micro-organisms, and since the bags and baskets usually employed do not prevent absorption of moisture, the following experiments were made by packing the sugar in an impermeable material.

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* *Handelingen voor het 4de Congres te Semarang*, 104.

## Baskets.

| Disinfected. | | | | | | | | Not Disinfected. | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| No. | Original Polarization. | Original Polarization on 100 Dry Substance. | Polarization at the end on 100 Dry Substance. | Difference. | Reducing Sugar original on 100 Dry Substance. | Reducing Sugar at the end on 100 Dry Substance. | Difference. | No. | Original Polarization. | Original Polarization on 100 Dry Substance. | Polarization at the end on 100 Dry Substance. | Difference. | Reducing Sugar original on 100 Dry Substance. | Reducing Sugar at the end on 100 Dry Substance. | Difference. |
| I. | 97·45 | 97·89 | 98·14 | −0·25 | 1·20 | 1·21 | 0·01 | I. | 97·10 | 97·87 | 98·01 | −0·14 | 1·33 | 1·38 | 0·05 |
| II. | 96·50 | 97·38 | 97·34 | 0·04 | 1·54 | 1·15 | −0·39 | II. | 96·50 | 97·41 | 96·83 | 0·58 | 1·54 | 1·61 | 0·06 |
| III. | 97·60 | 98·07 | 97·60 | 0·47 | 1·10 | 1·54 | 0·53 | III. | 97·60 | 98·12 | 96·96 | 1·16 | 1·10 | 2·04 | 0·94 |
| IV. | 96·70 | 97·80 | 96·94 | 0·86 | 1·44 | 2·07 | 0·63 | IV. | 96·80 | 97·86 | 94·43 | 3·43 | 1·39 | 3·47 | 2·08 |
| V. | 97·50 | 97·95 | 97·19 | 0·76 | 1·24 | 1·76 | 0·52 | V. | 97·10 | 97·76 | 94·71 | 3·05 | 1·29 | 3·73 | 2·44 |
| VI. | 97·00 | 97·45 | 97·31 | 0·14 | 1·49 | 1·62 | 0·13 | VI. | 97·10 | 97·53 | 97·55 | −0·02 | 1·44 | 1·55 | 0·11 |
| VII. | 96·60 | 97·34 | 95·13 | 2·21 | 1·54 | 2·99 | 1·45 | VII. | 97·40 | 98·11 | 96·54 | 1·57 | 1·20 | 2·21 | 1·01 |
| VIII. | 97·40 | 98·20 | 97·53 | 0·67 | 1·00 | 1·43 | 0·43 | VIII. | 97·40 | 98·20 | 96·82 | 1·38 | 0·97 | 2·04 | 1·07 |
| IX. | 97·40 | 97·97 | 94·88 | 3·09 | 0·91 | 3·24 | 2·33 | IX. | 97·40 | 97·97 | 93·95 | 4·02 | 0·91 | 3·89 | 2·98 |
| X. | 96·70 | 97·00 | 96·41 | 0·59 | 1·40 | 2·02 | 0·62 | X. | 96·80 | 97·15 | 96·74 | 0·41 | 1·28 | 1·85 | 0·57 |
| XI. | 97·50 | 98·17 | 96·91 | 1·26 | 0·99 | 2·04 | 1·05 | XI. | 97·50 | 98·19 | 96·43 | 1·76 | 0·99 | 2·41 | 1·52 |
| XII. | 96·50 | 97·32 | 96·33 | 0·99 | 1·29 | 2·27 | 0·98 | XII. | 96·80 | 97·61 | 94·63 | 2·98 | 1·21 | 3·22 | 2·01 |
| XIII. | 97·70 | 98·11 | 97·54 | 0·57 | 1·24 | 1·47 | 0·23 | XIII. | 97·60 | 98·02 | 97·03 | 0·99 | 1·24 | 1·94 | 0·70 |
| XIV. | — | — | — | — | — | — | — | XIV. | 97·50 | 98·19 | 97·25 | 0·93 | 1·04 | 1·68 | 0·64 |
| XV. | 97·00 | 97·74 | 96·25 | 2·49 | 1·24 | 1·69 | 0·45 | XV. | 97·20 | 97·85 | 94·21 | 3·64 | 1·24 | 3·40 | 1·16 |
| XVI. | 97·20 | 97·69 | 97·72 | −0·03 | 1·18 | 1·47 | 0·25 | XVI. | 97·30 | 97·75 | 97·08 | 0·67 | 1·13 | 1·85 | 0·72 |
| XVII. | 97·50 | 97·89 | 95·74 | 2·15 | 1·13 | 2·72 | 1·59 | XVII. | 97·40 | 97·87 | 97·18 | 0·69 | 1·12 | 1·63 | 0·51 |
| XVIII. | 97·10 | 97·69 | 97·35 | 0·34 | 1·33 | 1·54 | 0·21 | XVIII. | 97·20 | 97·58 | 95·93 | 1·55 | 1·38 | 2·41 | 1·03 |
| XIX. | 97·10 | 97·71 | 97·45 | 0·26 | 1·21 | 1·43 | 0·21 | XIX. | 97·10 | 97·91 | 95·99 | 1·72 | 1·21 | 2·22 | 1·01 |
| Average | 97·14 | 97·80 | 96·88 | 0·92 | 1·25 | 1·87 | 0·62 | Average | 97·20 | 97·82 | 96·22 | 1·60 | 1·26 | 2·34 | 1·08 |

## Bags.

| | | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| XVI. | 97·30 | 97·94 | 96·88 | 1·06 | 1·10 | 1·94 | 1·84 | XVI. | 97·20 | 97·78 | 96·27 | 1·51 | 1·12 | 2·04 | 0·92 |
| XVI. | 98·70 | 98·90 | 97·54 | 1·36 | 0·62 | 1·54 | 1·92 | XVI. | 98·70 | 98·90 | 96·44 | 2·46 | 0·58 | 2·27 | 1·69 |
| XVII. | — | — | — | — | — | — | — | XVII. | 99·30 | 99·50 | 95·07 | 4·43 | 0·30 | 3·35 | 3·05 |
| XIX. | — | — | — | — | — | — | — | XIX. | 97·20 | 97·79 | 95·98 | 1·81 | 1·17 | 2·60 | 1·43 |
| Average | 98·00 | 98·42 | 97·21 | 1·21 | 0·86 | 1·74 | 0·88 | Average | 98·10 | 98·50 | 95·94 | 2·55 | 0·79 | 2·56 | 1·77 |

Sugar, which had already attracted some moisture from the atmosphere, was packed in bags lined with waxed paper, and placed in a warehouse together with the ordinary bags filled with the same sugar, which latter served as a check. The analyses at the commencement, and after some time, were as follows :—

| Analyses. | Single Bag. | Bags Lined with Thin Waxed Paper. | Bags Lined with Thick Waxed Paper. |
|---|---|---|---|
| *At the beginning.* | | | |
| Polarization | 96·70 | 96·70 | 96·70 |
| Reducing sugar | 1·48 | 1·48 | 1·48 |
| Ash | 0·45 | 0·45 | 0·45 |
| Water | 0·73 | 0·73 | 0·73 |
| Undetermined | 0·64 | 0·64 | 0·64 |
| Total | 100·— | 100·— | 100·— |
| Nett | 92·97 | 92·97 | 92·97 |
| *After 2½ months.* | | | |
| Polarization | 95·00 | 95·00 | 95·10 |
| Reducing sugar | 1·48 | 1·48 | 1·45 |
| Ash | 0·45 | 0·45 | 0·45 |
| Water | 2·12 | 2·14 | 2·12 |
| Undetermined | 0·95 | 0·93 | 0·88 |
| Total | 100·— | 100·— | 100·— |
| Nett | 91·27 | 91·27 | 91·30 |
| *After 4½ months.* | | | |
| Polarization | 88·60 | 86·70 | 90·10 |
| Reducing Sugar | 2·84 | 2·84 | 2·32 |
| Ash | 0·48 | 0·47 | 0·48 |
| Water | 3·91 | 5·17 | 3·14 |
| Undetermined | 4·17 | 4·82 | 3·96 |
| Total | 100·— | 100·— | 100·— |
| Nett | 83·36 | 81·51 | 85·68 |

A second experiment was carried out with bags lined with paper rendered impermeable by immersion in a rubber solution. The bags were filled on August 12th, and analysed on the 12th October, December, and February, with these results:—

| | August 12th. | October 12th. | December 12th. | February 12th. |
|---|---|---|---|---|
| Polarization .. .. | 97·80 | 97·10 | 96·10 | 95·0 |
| Reducing Sugar .... | 0·82 | 0·98 | 1·65 | 2·05 |
| Ash .. .. .. .. | 0·31 | 0·30 | 0·31 | 0·30 |
| Water .. .. .... | 0·52 | 0·78 | 1·18 | 1·34 |
| Undetermined .. .. | 0·55 | 0·84 | 0·76 | 1·31 |
| Nett.. .. .. .... | 95·43 | 94·62 | 92·90 | 91·45 |

The lined bags retained the syrupy liquid which gradually collected between the sugar crystals and the bags were externally clean, whereas the ordinary bags become damp and sticky from the sweating of the contents, but did not hinder the free passage of gases and vapours. The sugar attracted moisture from the atmosphere both in the lined and unlined bags and afterwards became inverted in the same way, to the same degree, and in the same period of time.

**Keeping Sugar Dry.** It being impossible to modify the packing material so that the absorption of moisture and development of micro-organisms may be prevented, the only alternative is to deliver the sugar dry and to store it in dry, well-constructed and ventilated stores. If this is feasible, the keeping power of the sugar is guaranteed, but if not, nothing will prevent it from absorbing moisture and deteriorating in the end.

Sugar crystals are not hygroscopic, but the surrounding film of molasses is so, especially if dried artificially. In order to determine the hygroscopic power at various temperatures and moisture conditions of the atmosphere, quantities of 100 grms. of different qualities of sugar were spread out in porcelain dishes and placed in a room the temperature and humidity of which could be regulated. The dishes were weighed several times each day and the temperature and humidity of the air recorded.

ORIGIN AND ANALYSIS OF THE SUGARS.

| No. | Description of the Sugars. | Polarization. | Reducing Sugars. | Ash. | Water. |
|---|---|---|---|---|---|
| 1 | Refined sugar from Europe .. .. .. .. .. | 99·7 | — | — | 0·05 |
| 2 | Sugar over No. 25 D.S. from a carbonating factory | 99·6 | — | — | 0·05 |
| 3 | ,, ,, defecating ,, | 99·65 | — | — | 0·10 |
| 4 | Refining Crystals, No. 14 D.S. .. .. .. .... | 98·1 | 0·37 | 0·17 | 0·62 |
| 5 | ,, No. 11 ,, .. .. .. .. .. | 98·2 | 0·48 | 0·18 | 0·40 |
| 6 | Refining crystals from a masse cuite of 80 quotient | 97·8 | 0·82 | 0·31 | 0·52 |
| 7 | ,, ,, ,, 72 ,, | 97·5 | 0·87 | 0·20 | 0·50 |
| 8 | ,, ,, mixed masse cuite .. .. | 97·5 | 1·34 | 0·20 | 0·56 |
| 9 | Molasses sugar from a masse cuite of 62 quotient.. | 85·3 | 4·46 | 1·27 | 2·66 |
| 10 | The same but very finely grained .. .. .... | 84·8 | 1·95 | 1·67 | 3·70 |
| 11 | First sugar (carbonatation), No. 18 D.S. .. .. | 99·1 | 0·37 | 0·13 | 0·14 |
| 12 | ,, ,, No. 16 D.S.. .... | 98·8 | 0·62 | 0·14 | 0·32 |
| 13 | ,, (defecation) No. 20 D.S. .. .. | 98·9 | 0·55 | 0·14 | 0·26 |
| 14 | ,, ,, No. 18 D.S. .... | 98·4 | 0·92 | 0·16 | 0·38 |
| 15 | ,, ,, No. 16 D.S. .. .. | 98·1 | 0·71 | 0·14 | 0·56 |
| 16 | Second sugar (carbonatation), No. 20 D.S. .... | 98·2 | 0·54 | 0·16 | 0·68 |
| 17 | ,, (defecation) No. 18 D.S. .. .. | 95·2 | 1·20 | 0·16 | 2·88 |
| 18 | ,, ,, No. 10 D.S. .... | 91·5 | 3·50 | 0·91 | 2·44 |

The following tables contain the readings of dry and wet bulb thermometers with the corresponding values for absolute and relative humidity of the atmosphere, and the loss or increase in weight of the sugars, taking their original weights as 100. A decrease is shown by the sign —

| Date. | Hour. | Thermometer. | | Humidity. | | 1. | 2. | 3. | 4. | 5. | 6. | 7. | 8. | 9. | 10. |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Dry Bulb. | Wet Bulb. | Absolute. | Relative. | | | | | | | | | | |
| 22nd July. | 10·5 | 26·2 | 20·4 | 18·0 | 72 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| ,, ,, | 3 | 26·0 | 23·0 | 19·0 | 76 | 0 | 0 | 0 | 0 | 0·4 | 0·7 | 0·7 | 0·5 | 0·3 | 0·7 |
| ,, ,, | 5 | 25·8 | 23·0 | 19·2 | 78 | 0 | 0·1 | 0 | 0 | 0·4 | 0·7 | 0·7 | 0·2 | 0·3 | 0·7 |
| 23rd ,, | 7·5 | 24·0 | 21·8 | 18·0 | 81 | 0·3 | 0·3 | 0·2 | 0·3 | 1·2 | 2·2 | 2·0 | 0·8 | 1·7 | 2·7 |
| ,, ,, | 12 | 26·6 | 24·4 | 21·4 | 82 | 0·2 | 0·2 | 0·2 | 0·3 | 0·6 | 1·7 | 1·8 | 0·5 | 1·5 | 2·2 |
| ,, ,, | 3 | 25·8 | 28·4 | 19·9 | 81 | 0·3 | 0·2 | 0·1 | 0·3 | 0·6 | 1·7 | 1·7 | 1·5 | 1·5 | 2·2 |
| 24th ,, | 7·5 | 24·8 | 28·0 | 19·8 | 85 | 0·3 | 0·2 | 0·2 | 0·5 | 1·4 | 2·7 | 3·0 | 2·2 | 3·4 | 4·2 |
| ,, ,, | 12 | 27·2 | 28·8 | 19·8 | 73 | 0 | 0·1 | 0·2 | 0·2 | 1·2 | 2·5 | 3·0 | 1·5 | 3·4 | 4·2 |
| .. ,, | 3 | 26·6 | 24·2 | 21·0 | 83 | 0·2 | 0·5 | 0·3 | 0·2 | 1·2 | 2·7 | 3·0 | 1·7 | 3·8 | 4·2 |
| ,, ,, | 5 | 28·0 | 24·6 | 20·9 | 74 | 0·2 | 0·2 | —0·1 | 0·2 | 0·9 | 2·7 | 2·8 | 0·7 | 3·5 | 4·2 |
| 25th ,, | 7·5 | 26·0 | 23·4 | 19·8 | 79 | 0·2 | 0·2 | 0 | 0·3 | 1·0 | 2·2 | 2·7 | 0·5 | 3·5 | 4·4 |
| ,, ,, | 10·5 | 27·7 | 23·6 | 19·1 | 69 | 0·1 | 0 | 0 | 0·2 | 0·8 | 1·5 | 2·5 | 0 | 3·3 | 4·1 |
| ,, ,, | 12·5 | 31·1 | 25·4 | 20·6 | 61 | 0 | 0 | —0·1 | 0 | 0·6 | 1·2 | 2·5 | 0 | 2·8 | 3·7 |
| ,, ,, | 3 | 32·0 | 26·0 | 21·3 | 60 | 0 | 0 | —0·1 | 0 | 0·4 | 0·7 | 2·0 | 0 | 2·3 | 3·0 |
| ,, ,, | 5 | 30·6 | 24·8 | 19·7 | 61 | 0 | 0 | —0·1 | 0 | 0 | 0·7 | 1·2 | 0 | 1·3 | 2·2 |
| 26th ,, | 7·5 | 28·0 | 24·4 | 20·5 | 72 | 0 | 0 | —0·1 | 0 | 0 | 0·5 | 1·3 | 0 | 1·1 | 1·8 |
| ,, ,, | 12 | 30·1 | 25·0 | 20·4 | 64 | 0 | 0 | —0·1 | —0·1 | 0 | 0·3 | 1·0 | 0 | 1·0 | 1·6 |
| ,, ,, | 3 | 31·2 | 25·4 | 20·7 | 61 | 0 | 0 | —0·1 | —0·2 | —0·1 | 0·2 | 0·7 | —0·1 | 0·7 | 1·2 |

| Date. | Hour | Thermometer. | | Humidity. | | 11 | 12 | 13 | 14 | 15 | 16 | 17 | 18 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Dry Bulb. | Wet Bulb. | Absolute. | Relative. | | | | | | | | |
| 26th August. | 8 | 28·4 | 24·8 | 21·0 | 73 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| ,, ,, | 12 | 30·2 | 25·2 | 20·7 | 65 | 0 | 0 | 0 | —0·2 | —0·2 | —0·3 | —0·3 | —0·2 |
| ,, ,, | 3 | 30·7 | 25·4 | 20·8 | 63 | —0·2 | —0·2 | —0·2 | —0·3 | —0·3 | —0·7 | —0·5 | —0·3 |
| ,, ,, | 5 | 30·6 | 25·4 | 20·7 | 63 | —0·2 | —0·2 | —0·1 | —0·4 | —0·4 | —1·0 | —1·2 | —0·5 |
| 27th ,, | 7·5 | 27·2 | 25·0 | 20·2 | 83 | 0 | 0·3 | 0·3 | 0·2 | 0·2 | —0·5 | —1·2 | 1·3 |
| ,, ,, | 3 | 30·1 | 25·6 | 21·6 | 68 | —0·1 | 0·3 | 0·3 | —0·1 | —0·2 | —0·7 | —2·5 | 1·5 |
| ,, ,, | 5 | 29·0 | 24·6 | 20·3 | 68 | —0·2 | 0·2 | 0·2 | —0·2 | —0·2 | —1·0 | —2·8 | 1·1 |
| 28th ,, | 7·5 | 26·6 | 23·8 | 20·2 | 78 | 0 | 0·7 | 0·3 | 0 | 0 | —0·5 | —2·0 | 3·1 |
| ,, ,, | 3 | 30·6 | 25·4 | 20·9 | 64 | —0·2 | 0·3 | 0·1 | —0·3 | —0·3 | —1·0 | —2·3 | 2·1 |
| ,, ,, | 7·5 | 27·2 | 24·4 | 21·0 | 78 | 0·5 | 0·3 | 0·3 | 0 | 0 | —0·8 | —2·5 | 2·1 |
| 29th ,, | 12 | 27·2 | 24·8 | 21·8 | 81 | 0·3 | 0·4 | 0·3 | 0 | 0 | —1·0 | —2·4 | 3·8 |
| ,, ,, | 5 | 27·4 | 25·0 | 20·1 | 81 | 0·3 | 0·5 | 0·3 | 0 | 0 | —1·0 | —2·4 | 3·8 |
| 30th ,, | 7·5 | 25·8 | 24·0 | 21·0 | 85 | 0·4 | 0·8 | 1·0 | 0·5 | 0·5 | —0·2 | —1·2 | 6·3 |
| ,, ,, | 12 | 27·0 | 23·4 | 19·6 | 74 | 0·3 | 0·6 | 0·7 | 0·2 | 0·2 | —0·6 | —1·6 | 6·0 |
| 31st ,, | 7·5 | 26·2 | 24·6 | 22·0 | 87 | 0·5 | 0·8 | 1·0 | 0·3 | 0·2 | —0·5 | —1·3 | 7·0 |

When the experiment commenced, the sugars had already absorbed the full amount of moisture corresponding with the humidity and temperature of the atmosphere at that time.

The experiments indicate that pure sugar is not hygroscopic and contains very little moisture, whereas sugar crystals coated with molasses can absorb a considerable amount of moisture and thereby favour the development of micro-organisms. The figures for decrease or increase in moisture content indicate that it is the relative rather than the absolute humidity of the air which influences the absorption of moisture.

It should be remembered that the absolute humidity is the tension of the water vapour in the air expressed in mm. of mercury, while the relative humidity is the relation between that figure and the tension of the water vapour at that same temperature when the air is fully saturated with water. This latter is taken as a base, with the figure 100.

It is, therefore, not the tension of the aqueous vapour which determines the absorption or evaporation of water but the degree of saturation of the atmosphere with water vapour, whereby a given sugar absorbs water and then gives it off again at different temperatures but with the same tension.

The tables show that at temperatures between 24 and 33° C., refined sugar and very high grade raw sugars absorb moisture at a relative humidity of 80 and over, and give it off under that figure. The limit may be put down at 75 for white first sugars, at 70 for refining crystals and molasses sugar, and between 70 and 75 for seconds, according to their quality. It made no difference whether the refining crystals were dark coloured or light, finely or coarsely grained, made from pure syrup or from mixed syrup masse cuite and returned molasses. The only active condition is the percentage of adhering molasses.

We thus find that although pure sugar practically does not absorb moisture, raw sugar is hygroscopic and, however well-dried, will absorb moisture from the air as soon as it finds an opportunity to do so, and that the packing material is unable to prevent this.

This opportunity arises as soon as the sugar comes in contact with air containing over 70-75 relative humidity at temperatures prevailing in the tropics.

The meteorological observations made at the West Java Sugar Experimental Station show that in many cases those humidities exceed the figures mentioned.

Observations made at Kagok (Residency of Tegal), Island of Java, 109° 12′ 30″ E. Long., 6° 95′ 10″ Lat., 38·5 m. above sea level.

AVERAGE TEMPERATURE IN C.°.

| Year. | Jan. | Feb. | March. | April. | May. | June. | July. | August. | Sept. | October | Nov. | Dec. | Average |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1889 | 26·9 | 26·8 | 26·8 | 27·5 | 27·3 | 26·4 | 26·0 | 26·2 | 26·5 | 27·3 | 27·1 | 26·8 | 26·8 |
| 1890 | 26·8 | 26·3 | 26·5 | 26·7 | 26·2 | 25·9 | 25·6 | 25·4 | 26·2 | 26·4 | 26·3 | 26·2 | 26·2 |
| 1891 | 26·2 | 25·9 | 26·3 | 26·6 | 26·6 | 25·9 | 25·3 | 25·4 | 27·0 | 27·9 | 26·9 | 27·0 | 26·4 |
| 1892 | 25·7 | 26·2 | 26·4 | 26·2 | 26·2 | 26·0 | 25·8 | 26·0 | 26·5 | 26·7 | 26·1 | 26·2 | 26·2 |
| 1893 | 25·4 | 25·4 | 25·9 | 26·4 | 26·5 | 26·0 | 25·9 | 26·2 | 26·6 | 26·8 | 26·3 | 26·0 | 26·1 |
| 1894 | 25·7 | 25·7 | 25·9 | 26·3 | 25·9 | 25·4 | 25·5 | 25·7 | 26·3 | 27·3 | 26·3 | 26·2 | 26·0 |
| 1895 | 25·6 | 25·9 | 26·1 | 26·5 | 26·6 | 26·2 | 25·5 | 25·5 | 26·8 | 27·1 | 26·8 | 26·1 | 26·2 |
| 1896 | 26·1 | 25·9 | 26·2 | 26·2 | 26·3 | 25·6 | 25·5 | 26·1 | 26·8 | 27·9 | 27·6 | 26·4 | 26·4 |
| 1897 | 26·9 | 26·2 | 26·9 | 27·0 | 26·9 | 26·9 | 26·2 | 26·2 | 27·3 | 28·0 | 27·2 | 26·8 | 26·9 |
| 1898 | 26·2 | 26·4 | 26·4 | 26·6 | 26·6 | 26·1 | 25·8 | 26·0 | 26·7 | 26·9 | 26·7 | 26·5 | 26·4 |
| Average | 26·1 | 26·1 | 26·3 | 26·5 | 26·6 | 26·1 | 25·7 | 25·9 | 26·7 | 27·2 | 26·7 | 26·4 | 26·4 |

## Average Absolute Humidity of the Air in mm. Mercury.

| Year. | Jan. | Feb. | March. | April. | May. | June. | July. | August. | Sept. | October | Nov. | Dec. | Average |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1889 | 21·8 | 22·0 | 22·0 | 22·5 | 21·9 | 21·4 | 19·8 | 18·3 | 19·4 | 20·2 | 20·2 | 20·0 | 20·8 |
| 1890 | 20·8 | 20·6 | 21·2 | 20·6 | 20·7 | 20·2 | 18·9 | 18·0 | 18·1 | 19·1 | 20·1 | 20·3 | 19·9 |
| 1891 | 20·7 | 20·7 | 20·8 | 20·8 | 19·0 | 19·2 | 16·0 | 15·2 | 16·2 | 17·2 | 19·3 | 20·5 | 18·0 |
| 1892 | 21·2 | 21·4 | 21·9 | 21·6 | 20·0 | 19·5 | 19·3 | 19·4 | 18·5 | 19·9 | 20·3 | 20·1 | 20·2 |
| 1893 | 20·5 | 20·8 | 20·7 | 20·9 | 21·0 | 20·8 | 19·4 | 19·1 | 18·9 | 20·0 | 20·4 | 20·6 | 20·2 |
| 1894 | 21·2 | 21·3 | 21·2 | 21·0 | 19·9 | 19·3 | 18·4 | 16·8 | 19·9 | 19·8 | 20·1 | 21·4 | 20·0 |
| 1895 | 21·2 | 21·7 | 21·4 | 21·4 | 21·6 | 21·7 | 21·3 | 19·0 | 17·6 | 19·3 | 20·5 | 21·6 | 20·7 |
| 1896 | 21·9 | 22·1 | 21·8 | 22·1 | 22·1 | 18·0 | 17·2 | 16·9 | 16·7 | 18·3 | 20·2 | 22·0 | 19·8 |
| 1897 | 24·5 | 22·4 | 22·2 | 22·1 | 20·4 | 19·2 | 19·2 | 17·2 | 17·8 | 19·7 | 20·3 | 20·7 | 20·5 |
| 1898 | 21·5 | 22·1 | 21·4 | 21·7 | 20·8 | 20·2 | 19·2 | 18·5 | 19·1 | 20·1 | 20·3 | 21·1 | 20·5 |
| Average | 21·5 | 21·5 | 21·5 | 21·5 | 20·6 | 20·0 | 18·9 | 17·8 | 18·2 | 19·4 | 20·2 | 20·8 | 20·1 |

## Average Relative Humidity of the Air.

Full Saturation = 100.

| Year. | Jan. | Feb. | March. | April. | May. | June. | July. | August. | Sept. | October | Nov. | Dec. | Average |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1889 | 83 | 85 | 84 | 83 | 82 | 84 | 80 | 74 | 76 | 76 | 77 | 77 | 81 |
| 1890 | 80 | 82 | 83 | 80 | 82 | 82 | 79 | 77 | 73 | 76 | 80 | 81 | 80 |
| 1891 | 82 | 84 | 82 | 81 | 74 | 77 | 69 | 65 | 63 | 64 | 72 | 79 | 74 |
| 1892 | 86 | 85 | 86 | 86 | 80 | 78 | 79 | 78 | 73 | 78 | 81 | 80 | 81 |
| 1893 | 85 | 87 | 84 | 82 | 84 | 84 | 79 | 76 | 79 | 77 | 88 | 83 | 82 |
| 1894 | 87 | 87 | 85 | 82 | 82 | 81 | 76 | 59 | 77 | 75 | 80 | 87 | 80 |
| 1895 | 87 | 88 | 85 | 84 | 84 | 86 | 87 | 78 | 69 | 73 | 78 | 86 | 79 |
| 1896 | 87 | 89 | 86 | 88 | 81 | 75 | 72 | 68 | 65 | 67 | 75 | 86 | 76 |
| 1897 | 84 | 88 | 85 | 84 | 78 | 74 | 77 | 69 | 67 | 74 | 77 | 80 | 79 |
| 1898 | 84 | 87 | 83 | 84 | 81 | 85 | 78 | 78 | 77 | 77 | 78 | 82 | 81 |
| Average | 85 | 86 | 84 | 83 | 81 | 81 | 78 | 71 | 72 | 74 | 79 | 82 | 79·3 |

Observations made at Pekalongan, Java, 109° 40′ 26″ E. Long., 6° 52′ 39″ S. L., 4 m. above sea level.

## Average Temperature °C.

| Year. | Jan. | Feb. | March. | April. | May. | June. | July. | August. | Sept. | October | Nov. | Dec. | Average |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1902 | 26·49 | 25·91 | 26·43 | 27·96 | 27·47 | 26·68 | 25·94 | 26·33 | 26·75 | 27·92 | 28·70 | 27·54 | 27·01 |
| 1903 | 27·73 | 26·67 | 27·07 | 27·36 | 27·21 | 26·78 | 26·39 | 26·83 | 27·30 | 27·81 | 26·99 | 26·10 | 27·02 |
| 1904 | 26·07 | 25·93 | 26·56 | 26·83 | 26·70 | 26·39 | 26·16 | 26·29 | 26·99 | 27·38 | 26·83 | 26·29 | 26·54 |
| 1905 | 26·64 | 26·07 | 27·26 | 26·90 | 27·05 | 27·10 | 25·93 | 26·19 | 26·86 | 27·82 | 27·56 | 27·53 | 26·91 |
| 1906 | 26·41 | 27·25 | 27·10 | 27·02 | 27·28 | 26·71 | 26·82 | 26·88 | 26·88 | 27·43 | 26·47 | 26·42 | 26·89 |
| Average | 26·67 | 26·64 | 26·69 | 27·21 | 27·14 | 26·73 | 26.25 | 26·50 | 26·96 | 27·67 | 27·31 | 26·98 | 26·87 |

AVERAGE ABSOLUTE HUMIDITY IN MM. MERCURY.

| Year. | Jan. | Feb. | March. | April. | May. | June. | July. | August. | Sept. | October | Nov. | Dec. | Average |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1902 | 22·79 | 22·46 | 22·45 | 22·98 | 21·49 | 20·31 | 19·45 | 19·99 | 19·26 | 20·34 | 21·39 | 22·39 | 21·27 |
| 1903 | 22·83 | 22·87 | 22·23 | 22·83 | 22·73 | 21·37 | 21·47 | 20·60 | 21·30 | 21·90 | 21·83 | 21·43 | 21·95 |
| 1904 | 22·22 | 21·90 | 22·13 | 22·24 | 22·67 | 22·09 | 20·62 | 20·86 | 21·66 | 22·78 | 22·67 | 22·27 | 22·07 |
| 1905 | 22·25 | 21·87 | 22·45 | 22·19 | 23·97 | 22·13 | 19·90 | 19·95 | 20·90 | 21·95 | 22·28 | 22·80 | 21·88 |
| 1906 | 22·57 | 23·10 | 22·57 | 22·50 | 22·40 | 21·20 | 21·47 | 21·47 | 22·07 | 21·97 | 22·27 | 22·40 | 22·25 |
| Average | 22·53 | 22·44 | 22·37 | 22·55 | 22·65 | 21·42 | 20·58 | 20·57 | 21·04 | 21·99 | 22·09 | 22·26 | 21·88 |

AVERAGE RELATIVE HUMIDITY. Full Saturation = 100.

| Year. | Jan. | Feb. | March. | April. | May. | June. | July. | August | Sept. | October | Nov. | Dec. | Average |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1902 | 90 | 91 | 89 | 83 | 79 | 78 | 79 | 79 | 75 | 74 | 71 | 82 | 80·8 |
| 1903 | 83 | 88 | 84 | 85 | 85 | 82 | 85 | 79 | 80 | 80 | 83 | 86 | 83·3 |
| 1904 | 89 | 88 | 86 | 84 | 84 | 86 | 86 | 83 | 82 | 84 | 85 | 88 | 85·4 |
| 1905 | 86 | 87 | 84 | 85 | 84 | 83 | 81 | 80 | 80 | 79 | 89 | 84 | 83·5 |
| 1906 | 89 | 86 | 85 | 85 | 84 | 83 | 83 | 82 | 84 | 81 | 87 | 88 | 84·6 |
| Average | 87·4 | 88·0 | 85·6 | 84·4 | 83·2 | 81·2 | 81·4 | 80·8 | 80·2 | 79·6 | 83·0 | 85·6 | 82·5 |

In order to store sugar in good condition in moist, tropical regions, it must be exposed to the atmosphere no more than is absolutely necessary, by heaping up the bags of sugar in large and close piles, and by regulating the ventilation of the storehouse in such a way that the air is as little charged with water vapour as possible. To this end, hygrometers should be placed inside and outside the storehouses, so that one can know when to ventilate the sugar, and when to keep the doors closed and exclude the free admission of moist air.

CHAPTER V.

# EXHAUSTED MOLASSES.

## I.—Definition and Formation of Molasses.

On curing the final masse-cuites, a syrupy liquid is obtained, which, on further concentration and cooling, does not give any more crystals, and bears the name of "exhausted molasses." Although no sugar can be crystallized out, it always contains (in conjunction with the accumulated impurities originating from the clarified juice) a considerable amount of sucrose, which is therefore practically lost. This is due to the fact that the sucrose in the molasses no longer exists in a free state, but is combined with some constituents of the juice to form viscous, uncrystallizable bodies, which, on concentration, may be changed into a dry mass, but even then do not give up their sucrose. Unless we succeed in removing from the juice everything but sucrose and water during clarification, which is an impossibility, we are sure to find molasses at the end of every process of sugar extraction; and systems which claim to make sugar without any molasses can, without any further investigation, be deemed unreliable.*

**Definition.**

**Impossibility of Avoiding Molasses.**

The nature and conditions of molasses formation have for a long time been a point of dispute between different investigators, and even now the question is not yet definitely settled, although unanimity exists as regards the principal points.

Formerly it was the general opinion that sucrose could not crystallize from the final molasses because the non-saccharine constituents made the liquid so viscous at high concentrations that the sucrose molecules were prevented from combining into crystals, and were thus forced to remain in solution. At first sight, this explanation appears very plausible, but its probability decreases if we remember that sucrose crystallizes slowly yet perfectly from very stiff jellies, and a sucrose crystal can be made to grow regularly when suspended in a solution of sugar in isinglass.

**Mechanical Theory.**

**Objections against it.**

Further, this theory is at variance with the fact that sugar crystallizes from an osmosed molasses (where the non-diffusing viscous colloids are accumulated), whereas, according to the theory, crystallization should have totally ceased.

The so-called "mechanical theory" was soon abandoned, and the opinion was formed that the prevention of crystallization was due to chemical influences. A general idea prevailed that the sucrose combined with other constituents of the molasses to form very soluble bodies, so that

**Chemical Molasses Theory.**

* The so-called "concrete sugar" is only an apparent exception to this rule, as it contains the whole of the dry substance of the juice, crystals, and molasses in one agglomerated mass, and is therefore not a pure crystallized sugar.

the solubility of the sucrose and of the accompanying constituent was much greater than when both were present separately. According to Dubrunfaut, a solution of common salt dissolves more sucrose than the water contained in the solution would do, if it did not contain the salt. On the other hand, a saturated sucrose solution dissolves twice as much salt as the water contained in the solution would alone have done. Assuming that sucrose and sodium chloride enter into a very soluble combination, we can easily explain the formation of molasses, and if this simple conclusion from Dubrunfaut's experiment had been made sooner, it would have saved much misunderstanding.

**Mutual Increase or Decrease of Solubility between Sucrose and Salts.**

In beetroot molasses, the solubility of sucrose in the contained water is greater than in pure water at the same temperature, and as in former times beetroot molasses was the sole object of investigation, the idea arose that the solubility of sucrose is always higher than the normal in every molasses, and all theories and observations were based on that principle. Whilst it was known that some salts increased (as was the saying) the solubility of sucrose, and therefore possessed a melassigenic power, a great number of substances were tested as to their property of increasing or decreasing the solubility of sucrose in water, and thus determining their melassigenic power. Under the impression that the molasses-forming power was always identical with the increase in the solubility of sucrose, the different bodies were divided into positive, negative and indifferent molasses-formers, according to the increase, decrease, or stability of the solubility of sucrose in aqueous solution in the presence of those bodies. This division is still in great favour in most treatises and publications, and the denomination of *positive or negative molasses-formers* is still widely used. It was, however, observed that some bodies proved to be negative molasses-formers in dilute concentrations, but positive in high concentrations, and finally the subject led to a mass of misunderstanding and controversy.

**So-called Positive, Indifferent, and Negative Molasses-formers.**

The simplest explanation assumes that all salts, both organic and inorganic, combine with sucrose to form hydrated bodies, each having its own solubility. If we first consider the case where the solution contains as much sucrose and salt as possible, we observe two alternatives. Either the combination is very soluble in water, much salt and sucrose enters into solution, and we may call the salt a positive molasses-former; or the combination is only slightly soluble, the saturated solution therefore only contains relatively little salt and sucrose, and the salt thus belongs to the negative molasses-formers.

**Simple Combination of Sucrose and Salts.**

Köhler* showed that, in general, the solubility of sucrose in a solution of non-sugar, and also the solubility of the non-sugar in a sucrose solution, are mutually in such a relation, that from either body as much is dissolved as it can

* *Zeitschrift Rübenzuckerind.*, 47, 441.

dissolve of the other body, and that solutions containing sucrose and large quantities of non-sugar contain (with the same proportion of water), sometimes more and sometime less sucrose than corresponds with the solubility of pure sucrose in pure water. The most evident explanation is that, when present in an excess of both sucrose and salt, water can dissolve so much of both as corresponds with the solubility of the new combination formed between the sucrose and the salt. According to that solubility, the water can dissolve more or less combined sucrose than if the salt had not been present and the sucrose had remained in the uncombined state.

Köhler investigated how much salt was dissolved at the temperature of 31·25° C. in 100 c.c. of pure water, and how much in 100 c.c. of a sucrose solution saturated at that same temperature, and finally how many grammes of sucrose were contained in 100 c.c. of this salt solution.

| Name of the Salts. | Grammes of Salt in 100 c.c. of pure water. | Grammes of Salt in 100 c.c. of Saturated Sucrose Solution. | Grammes of Sucrose in 100 c.c. of the Salt Solution. |
|---|---|---|---|
| Potassium Chloride | 38·2 | 44·8 | 246·5 |
| Potassium Carbonate | 95·9 | 105·4 | 265·4 |
| Potassium Acetate | 286·3 | 293·5 | 324·8 |
| Potassium Citrate | 159·7 | 219·0 | 303·9 |
| Sodium Chloride | 35·9 | 42·3 | 236·3 |
| Sodium Carbonate | 22·0 | 24·4 | 229·2 |
| Sodium Acetate | 46·9 | 57·3 | 237·6 |
| Potassium Sulphate | 12·4 | 10·4 | 219·0 |
| Potassium Nitrate | 47·4 | 41·9 | 224·7 |
| Sodium Sulphate | 45·4 | 30·5 | 183·7 |
| Calcium Acetate | 35·4 | 26·3 | 190·3 |
| Calcium Chloride | 88·5 | 79·9 | 135·1 |
| Magnesium Sulphate | 47·5 | 36·0 | 119·6 |

**Solubility of the Combinations.**

As, in every case, a decreased solubility of the salt coincides with a lower solubility of the sucrose, it is evident that in every instance, where both the components occur in such large quantities that they can combine together to their full extent, the solubility of the constituents is regulated by that of their combination, and no longer by that of the respective bodies in an uncombined state. In connection with this point Degener* found that the lowest solubilities of salt and sucrose in solutions of these two in water correspond with the formulæ of $NaCl + 4\ C_{12}H_{22}O_{11}$ for the sodium chloride combination, and with $CaCl_2 + 3\ C_{12}H_{22}O_{11}$ for the corresponding calcium chloride combination.

**Solubility of Sucrose.**

In the case of one of the constituents being deficient, it is clear that no more of the combination is formed than corresponds with the amount of that constituent, so that we can no longer find the solubility of the combination only, but have now to take into account the amount of the still uncombined portion of the other constituent. If it is the salt

* *Deutsche Zuckerindustrie*, 20, 2149.

which is deficient, then there exists in the solution, besides the sucrose in combination, a certain amount of free sucrose, the solubility of which may not be neglected although it may be influenced by the presence of the combination. As these combinations are hydrated bodies, we can suppose the solution to consist of free water, free sucrose, combined water and a sucrose-salt combination. If we wished to ascertain whether the presence of a salt influences the solubility of sucrose in water and in which direction, and with that object only took into account the total amount of sucrose and the total amount of water, we can easily imagine what highly varying results might accrue, according to the different proportions in which the four constituents mutually exist.

When the amount of salt is small and that of sucrose considerable, only a little of the hydrated sucrose-salt combination will be formed, and very little free sucrose and free water will be withdrawn from the solution but sufficient to prevent the remaining free water from keeping all of the remaining free sucrose in solution, part of the latter crystallizing. We have, therefore, in the remaining portion of free water its full content of sucrose, and in the hydrate a little combined sucrose, and if the sum of these two quantities happens to be smaller than corresponds with the figure for the solubility of sucrose in water, the total amount of sugar in the total amount of water is smaller than the normal solubility, and we should call it a negative molasses-former. When the amount of salt increases, then of course the quantity of salt-sucrose combination increases equally, until at the end, all the sucrose has combined with salt, and we no longer have to deal with a solution of sucrose in a decreasing amount of free water, but only the solubility of a sucrose-salt combination, which is, as we saw before, always higher than that of the sucrose existing in it. This explains why in Herzfeld's experiments* additions of small portions of non-sugar were apt to lower the solubility of the total sucrose in the total water, whereas larger portions increased it.

In pure juices and syrups we see instances of the former case; in these products the amount of the combination is far less than the free sucrose, and accordingly the solubility of the total sucrose in the total water should be lower rather than higher than the normal figure. In the exhausted molasses, on the contrary, from which the superfluous sucrose has been removed by successive crystallizations, and only the saturated sucrose-salt combination left, it depends solely on the degree of concentration of this strongly evaporated product how much sucrose is found *on 100 parts* of water. In beetroot molasses, crystallized in large crystallizing tanks, this amount of water is generally such that the ratio of sucrose to water is greater than in a pure saturated sucrose solution, but, as we shall see later on, this is only a surmise; it might also have been the reverse. But, because beetroot molasses were studied almost exclusively, and in this substance the higher proportion was met with, the idea of supersaturated solutions has unconsciously taken root in the minds

**Cases from Practice.**

* *Zeitschrift Rübenzuckerind.*, 42, 182, 240.

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of all investigators who studied molasses and their formation, and they have always endeavoured to explain that supersaturation by the hypothesis of the presence of substances which prevented crystallization. A short definition of molasses (*i.e.*, of an ideal molasses) according to the author's theory is : *A hydrated syrupy-liquid combination of sucrose and salts.*

**Definition of an Ideal Molasses.**

After his experience of beetroot molasses, he was struck by the much smaller ratio of sucrose to water in cane molasses than corresponds with the solubility of sucrose in water, instead of the higher one he had always been led to suppose.

At the average Java temperature of about 28 C. one part of water can keep in solution 2·162 parts of sucrose, so that if that solubility was not changed by the presence of other bodies in the molasses, the polarization of a molasses with 25 per cent. of water should be 53·9. According to the following table of analyses of mother-liquors crystallized in tanks, the ratio of sucrose to water is generally less than that of the two pure ingredients, and generally those containing the most glucose show the lowest ratio :—

**Ratio of Sucrose to Water in Cane Molasses.**

| No. | Dry Substance. | Sucrose. | Reducing Sugar. | Ash. | Water. | Quotient of Purity. | Sucrose on 100 parts of water.* | Ratio of Reducing Sugar to Ash. |
|---|---|---|---|---|---|---|---|---|
| 0 | — | — | — | — | — | 100· | 216·2 | — |
| 1 | 74·88 | 59·20 | 7·29 | 6·07 | 25·12 | 79·06 | 235·7 | 1·20 |
| 2 | 74·29 | 58·70 | 9·97 | 3·82 | 25·71 | 79·01 | 228·3 | 2·61 |
| 3 | 82·88 | 47·80 | 14·53 | 6·66 | 17·12 | 57·67 | 279·2 | 2·18 |
| 4 | 74·69 | 50·20 | 15·14 | 4·35 | 25·31 | 67·21 | 198·3 | 3·48 |
| 5 | 82·90 | 43·70 | 15·80 | 6·50 | 17·10 | 52·17 | 255·6 | 2·43 |
| 6 | 72·23 | 49·00 | 16·52 | 2·52 | 27·77 | 67·84 | 176·4 | 6·56 |
| 7 | 83·55 | 47·90 | 17·44 | 7·00 | 16·45 | 57·33 | 291·2 | 2·49 |
| 8 | 71·07 | 42·90 | 19·93 | 3·00 | 28·93 | 60·36 | 148·3 | 6·64 |
| 9 | 71·55 | 43·10 | 20·87 | 3·39 | 28·45 | 60·23 | 151·5 | 6·16 |
| 10 | 77·10 | 34·10 | 23·00 | 7·80 | 22·90 | 44·23 | 148·9 | 2·95 |
| 11 | 71·40 | 32·40 | 23·00 | 5·20 | 28·60 | 45·38 | 113·3 | 4·42 |
| 12 | 76·80 | 36·90 | 26·30 | 4·97 | 23·20 | 48·05 | 159·1 | 5·29 |
| 13 | 75·86 | 30·90 | 27·47 | 6·37 | 24·14 | 40·73 | 128·0 | 4·31 |
| 14 | 73·88 | 32·90 | 27·53 | 3·72 | 26·12 | 44·53 | 126·0 | 7·40 |
| 15 | 80·90 | 35·30 | 27·60 | 8·08 | 19·10 | 43·63 | 184·8 | 3·42 |
| 16 | 74·62 | 34·05 | 27·78 | 4·16 | 25·38 | 45·63 | 134·2 | 6·68 |
| 17 | 75·50 | 33·70 | 28·12 | 3·65 | 24·50 | 44·64 | 137·6 | 7·70 |
| 18 | 77·02 | 33·80 | 28·20 | 6·88 | 22·98 | 43·88 | 147·1 | 4·10 |
| 19 | 73·78 | 32·19 | 30·30 | 3·77 | 26·22 | 43·64 | 122·8 | 8·04 |
| 20 | 74·23 | 30·69 | 31·73 | 3·65 | 25·77 | 41·34 | 119·1 | 8·69 |

* At 28° C.

This appears very strange, since, in existing literature, glucose is always referred to as a molasses-former, and one would expect high solubilities, instead of the low values found by actual analysis. Gunning* has said that glucose renders a certain amount of sucrose uncrystallizable. Pellet† put down the melassigenic coefficient of glucose as 0·60. Flourens‡ allows various values for that figure, fluctuating between 0·30 and 1·0, whilst Degener§ mentions having found glucose to possess no melassigenic power of its own, but able to transform sucrose into invert sugar by the action of its acid products of decomposition. In that connection Pellet† also mentions two distinct sources of molasses-forming by glucose, namely, one as a consequence of the inverting action of glucose, which he calculates as 0·16, and further the pure melassigenic power, at a value of 0·60. The alleged inverting action, which has, however, never been proved to exist, may be ruled out of consideration here, where only the melassigenic power itself is considered.

**Molasses-forming Property of Reducing Sugar.**

While many authors thus ascribe different values to the melassigenic power of glucose, the author has made some experiments to ascertain how this sugar influences the solubility of sucrose in water.

To this end solutions were made, each containing the same amount of sucrose and of water, but varying amounts of glucose. They consisted of 25 grms. of sucrose, 7·5 grms. of water, and 0, 0·5, 1, 3, 6, 12·5, and 25 grms. respectively of invert sugar or glucose.

The three constituents were dissolved by heat and put aside to cool, after the addition of a few sucrose crystals in order to promote crystallization of the supersaturated sucrose. After all the crystallizable sucrose had formed into crystals (a month being amply sufficient), the crystals were separated from the mother-liquor and both analysed. The result of these analyses was that in every case practically the same amount of sucrose had crystallized out, as is shown here.

| | 1. | 2. | 3. | 4. | 5. | 6. | 7. |
|---|---|---|---|---|---|---|---|
| Sucrose crystallized out.. | 9·3 | 9·1 | 10·0 | 8·9 | 9·8 | 9·2 | 9·0 |
| Sucrose dissolved.. .... | 15·7 | 15·9 | 15·0 | 16·1 | 15·2 | 15·8 | 16·0 |
| Invert sugar .. .. .. | 25·0 | 12·5 | 6·0 | 3·0 | 1·0 | ·5 | — |
| Water .. .. .. .... | 7·5 | 7·5 | 7·5 | 7·5 | 7·5 | 7·5 | 7·5 |

In all the mixtures, the amount of sucrose remaining dissolved in the water was therefore the same, showing that the solubility of sucrose in water in which already thrice its weight of glucose was dissolved was exactly the same as in water without any addition of glucose. In sample No. 1, 100 parts of water dissolved 210 parts of sucrose, while in No. 7, the solubility amounted to 213·3 parts in 100. It is evident that glucose has no influence whatever on the amount of sugar crystallized out.

* Saccharimetrie en accyns. † *Zeitschrift Rübenzuckerind.*, 1879, 806.
‡ *Journal des Fabriçants de Sucre*, 1880, 40. § *Zeitschrift Rübenzuckerind.*, 1881, 514.

He repeated the experiment and also determined the composition of the resulting mother liquor.

120 grms. of sucrose and 50 grms. of water were mixed, and to four of such mixtures were added 7·5, 15, 30, and 60 grms. of invert sugar respectively. The mixtures were raised to boiling-point until completely dissolved, sterilized in a Koch's sterilizer, and cooled. After that, two grms. of sucrose crystals were added to start crystallization, and the mixtures kept in a place where the temperature never rose above 29° C. After six weeks the samples were analysed with the following results. In the figure for the weight of sucrose crystallized out allowance is made for the two grms. of sugar added after cooling.

| Constituents. | Invert Sugar. | | | | |
|---|---|---|---|---|---|
| | 0. | 7·5. | 15. | 30. | 60. |
| *Weight in Grammes :—* | | | | | |
| Sucrose crystallized out .. .. | 14 | 14 | 15 | 15 | 14 |
| Sucrose dissolved .. . .... | 106 | 106 | 104 | 105 | 105 |
| Sucrose inverted .. .. .. .. | 0 | 0 | 1 | 0 | 1 |
| *Composition of the Mother-Liquor in %:* | | | | | |
| Sucrose .. .. .. .... | 67·6 | 64·1 | 60·9 | 56·2 | 48·7 |
| Invert Sugar .. .. .. .. .. | 0·1 | 5·3 | 9·7 | 16·7 | 27·5 |
| Ash .. .. .. .. .. .... | 0·1 | 0·1 | 0·1 | 0·2 | 0·2 |
| Water .. .. .. .. .. .. | 32·0 | 30·3 | 29·1 | 26·6 | 23·25 |
| Undetermined .. .. .. .... | 0·2 | 0·2 | 0·2 | 0·3 | 0·35 |
| Sucrose on 100 water .. .. .. | 211 | 212 | 209 | 211 | 210 |

The table shows very clearly that, contrary to the opinion of many authors on this subject, glucose or invert sugar does not affect the solubility of sucrose, and also that this sugar does not possess an inverting action. Neither can the other constituents of the molasses be made responsible for the observed lowering of the solubility of sucrose in cane sugar molasses, because they—organic and inorganic salts, pectin, gums, nitrogenous bodies, caramel, &c.,—are also found in beetroot molasses, and are there looked upon as causing the higher solubility of the sucrose.

**Melassigenic Power of the other Non-Sugars.**

As the joint action of invert sugar and salts might cause the phenomenon alluded to here, the author prepared solutions containing the same amount of sucrose and of water, but varying amounts of glucose and of an organic acid salt, potassium acetate.

**Joint Action of Invert Sugar and Salts.**

After the supersaturated sugar had crystallized out, it was weighed and the figures were as given underneath:—

| Constituents in grammes. | 1 | 2 | 3 | 4 | 5 | 6 | 7 |
|---|---|---|---|---|---|---|---|
| Total sucrose .. .. .. | 25 | 25 | 25 | 25 | 25 | 25 | 25 |
| Sucrose crystallized out.... | 9·2 | 9·1 | 6 | 3·1 | 11·6 | 10·7 | 6·7 |
| Sucrose dissolved .. .. | 15·8 | 15·9 | 19 | 21·9 | 13·4 | 14·3 | 18·3 |
| Glucose .. .. .. .... | — | — | — | — | 10 | 10 | 10 |
| Potassium acetate .. .. | — | 1 | 2 | 5 | 1 | 2 | 5 |
| Water.. .. .. .. .... | 7·5 | 7·5 | 7·5 | 7·5 | 7·5 | 7·5 | 7·5 |
| Sucrose on 100 water .. | 210·7 | 212·0 | 253·3 | 292·0 | 178·7 | 190·7 | 244·0 |

When repeating the experiment on a larger scale the mother-liquors were also analysed. The mixtures consisted of 120 or 150 grms. of sucrose, 30 grms. of invert sugar, 50 grms. of water, and 0, 1, 5, 10, 20, 30, and 40 grms. respectively of potassium acetate. After complete crystallization, the results were as follows:—

| Constituents. | Grammes of Potassium Acetate. | | | | | | |
|---|---|---|---|---|---|---|---|
| | 0 | 1 | 5 | 10 | 20 | 30 | 40 |
| *Weight in grammes:*— | | | | | | | |
| Total Sucrose.. .. .. | 120 | 120 | 120 | 120 | 120 | 150 | 150 |
| Sucrose crystallized out. | 15 | 45 | 25 | 16 | 3 | 18 | 6 |
| Sucrose dissolved .. .. | 105 | 75 | 94 | 104 | 115 | 132 | 144 |
| Sucrose inverted .... | — | — | 1 | — | 2 | — | — |
| *Composition of the liquid in per cent.:*— | | | | | | | |
| Sucrose .. .. .. .. | 56·2 | 48·0 | 52·9 | 53·8 | 53·4 | 54·6 | 53·8 |
| Glucose .. .. .... | 16·7 | 18·2 | 15·8 | 15·0 | 13·97 | 12·37 | 11·6 |
| Ash .. .. .. .. .. | 0·2 | 0·88 | 2·29 | 3·54 | 6·51 | 8·70 | 10·56 |
| Water .. .. .. .... | 26·6 | 32·07 | 28·11 | 25·81 | 23·21 | 20·78 | 18·10 |
| Undetermined.. .. .. | 0·3 | 0·85 | 0·90 | 1·75 | 2·91 | 3·55 | 5·94 |
| Glucose: Ash .. .... | — | 20·7 | 6·9 | 4·3 | 2·1 | 1·4 | 1·1 |
| Sucrose on 100 Water.. | 211 | 150 | 188 | 209 | 230 | 263 | 297 |

We see from the tables that glucose when unaccompanied by salts does not interfere with the solubility of the sucrose; that the organic acid salt (without addition of glucose) causes more sucrose to remain dissolved, but that the combination of glucose and organic salts strongly decreases the solubility of sucrose in case of a high ratio between glucose and ash, and increases the same when the ratio between glucose and ash is low.

In cane products these quotients are generally high; and this suggested further investigations under such conditions as occur in practical working.

**Experiments with Sucrose and Salts.**

120 grms. of sucrose were mixed with 50 grms. of water and as much salt as would represent about 4 grms. of ash, the quantity of glucose added to the mixtures being varied within rather wide limits. As representatives of the organic salts derived from the juice (malates and succinates), the acetates of sodium, potassium, and calcium were employed. To study the influence of those organic salts that are formed from glucose during the process of manufacture, a solution containing a large proportion of glucose was boiled with small portions of lime until almost all glucose reaction had disappeared from the solution. The glucose had become transformed into lime salts of organic acids, and all of the free lime had entered into that combination. The solution was then filtered and divided into three equal parts, of which one was evaporated to a syrupy liquid and represented the solutions of the lime salt. The two other portions were transformed into the corresponding sodium and potassium salts by decomposition with carbonate of sodium and of potassium. These two liquids were also filtered off and evaporated, and represented respectively a solution of the sodium and potassium glucinate. Their composition was as follows :—

| Lime Salt. | | Potassium Salt. | | Sodium Salt. | |
|---|---|---|---|---|---|
| CaO .. .. .. | 9·53 | $K_2O$ .. .. . | 17·67 | $Na_2O$ .. .. .. | 8·44 |
| Glucose .. .... | 3· | Glucose .. .... | 4· | Glucose .. .... | 3· |
| Organic acid .. | 44·07 | Organic acid .. | 42·23 | Organic acid .. | 34·16 |
| Water .. .... | 43·40 | Water .. .... | 36·10 | Water .. ... | 54·40 |
| | 100· | | 100· | | 100· |

From each of these solutions (which were very viscous) such a quantity was weighed out as would contain 4 grms. of ash, and the amounts of water and glucose were determined. In the tables given underneath those salts are referred to under the denomination of glucinates, although they are by no means chemically identified bodies. The following mixtures were prepared containing lime, sodium, and potassium salts, with and without addition of organic salts. When dissolving the salts it was observed that they dissolved much more slowly and more difficultly in the thick liquid containing sugars than they would have done in the same amount of pure water as was present in the syrups.

| Added to 120 grms. of Sucrose and 50 gr. of Water. | | |

The analytical and calculated figures here follow :—

| Constituents. | Potassium Acetate. | | | | | | Sodium Acetate. | | |
|---|---|---|---|---|---|---|---|---|---|
| | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 |
| *Weight in grammes :—* | | | | | | | | | |
| Sucrose crystallized out .. | 18·50 | 5·3 | 17·5 | 21·8 | 24·3 | 29·0 | 5·4 | 14·3 | 18·2 |
| Sucrose dissolved .. .... | 101·5 | 105·0 | 103·0 | 99·0 | 96·0 | 80·5 | 113·5 | 104·0 | 97·0 |
| Sucrose inverted.. .. .. | — | 9·7 | — | — | 4·7 | — | 1·1 | 1·7 | 4·8 |
| *Composition of the solution in per cent. :—* | | | | | | | | | |
| Sucrose .. .. .. .... | 64·20 | 62·20 | 61·30 | 56·60 | 52·00 | 42·30 | 62·60 | 53·20 | 45·30 |
| Invert Sugar .. .. .. | 0·80 | 5·31 | 6·20 | 11·50 | 16·70 | 29·10 | 5·20 | 17·30 | 27·80 |
| Ash .. .. .. .. .... | 2·50 | 2·10 | 2·01 | 2·07 | 2·08 | 1·80 | 2·36 | 2·24 | 1·77 |
| Water .. .. .. .. .. | 31·60 | 29·60 | 29·80 | 28·90 | 28·62 | 26·20 | 27·50 | 25·50 | 23·30 |
| Undetermined.. .. .... | 0·90 | 0·79 | 0·69 | 0·93 | 0·60 | 0·60 | 2·34 | 1·76 | 1·83 |
| Invert Sugar: Ash .. .. | 0·32 | 2·53 | 3·07 | 5·55 | 8·03 | 16·17 | 2·20 | 7·80 | 16·0 |
| Sucrose on 100 water .... | 203 | 210 | 206 | 198 | 182 | 161 | 227 | 208 | 194 |

| Constituents. | Calcium Acetate. | | | Potassium Glucinate. | | | Sodium Glucinate. | | |
|---|---|---|---|---|---|---|---|---|---|
| | 10 | 11 | 12 | 13 | 14 | 15 | 16 | 17 | 18 |
| *Weight in grammes :—* | | | | | | | | | |
| Sucrose crystallized out .. | 14·5 | 36·8 | 40·1 | 23·5 | 35·8 | 33·4 | 24·1 | 29·7 | 30·1 |
| Sucrose dissolved .. .... | 102·5 | 81·0 | 79·0 | 93·5 | 82·5 | 85·0 | 92·5 | 84·5 | 82·0 |
| Sucrose inverted.. .. .. | 3 | 2·2 | 0·0 | 3 | 1·7 | 1·6 | 3·4 | 5·8 | 7·8 |
| *Composition of the solution in per cent. :—* | | | | | | | | | |
| Sucrose .. .. .. .... | 61·50 | 48·80 | 40·90 | 57·80 | 48·40 | 41·80 | 57·60 | 48·60 | 40·40 |
| Invert Sugar .. .. .. | 4·30 | 18·30 | 30·04 | 7·10 | 17·60 | 29·60 | 7·31 | 19·20 | 30·80 |
| Ash .. .. .. .. .... | 2·02 | 1·78 | 1·44 | 2·10 | 2·13 | 1·76 | 1·85 | 1·62 | 1·44 |
| Water .. .. .. .. .. | 30·01 | 29·46 | 25·91 | 30·80 | 29·30 | 21·60 | 30·50 | 28·78 | 24·73 |
| Undetermined .. .... | 2·17 | 1·66 | 1·71 | 2·20 | 2·57 | 2·24 | 2·74 | 1·80 | 2·63 |
| Invert Sugar : Ash .. .. | 2·12 | 10·3 | 20·90 | 3·40 | 8·26 | 16·82 | 4·0 | 11·85 | 21·40 |
| Sucrose on 100 water .... | 205 | 162 | 158 | 187 | 165 | 170 | 185 | 169 | 164 |

| Constituents. | Calcium Glucinate. | | | Sodium Chloride. | | | |
|---|---|---|---|---|---|---|---|
| | 19 | 20 | 21 | 22 | 23 | 24 | 25 |
| *Weight in grammes :—* | | | | | | | |
| Sucrose crystallized out .. .. .. .. | 34·7 | 45·3 | 46·1 | 12·50 | 12·7 | — | — |
| Sucrose dissolved .. .. .. .. .... | 84·0 | 73·0 | 72·5 | 107 | 103·5 | 56·0 | 38·5 |
| Sucrose inverted .. .. .. .. .. .. | 1·8 | 1·7 | 1·4 | — | 3·8 | 64·0 | 81·5 |
| *Composition of the solution in per cent. :—* | | | | | | | |
| Sucrose .. .. .. .. .. .. .. .. | 56·50 | 45·30 | 37·90 | 66 20 | 62 2 | 27·20 | 16·70 |
| Invert Sugar .. .. .. .. .. .... | 4·49 | 18·60 | 31·40 | 0·40 | 4·80 | 46·30 | 59·70 |
| Ash .. .. .. .. .. .. .. .. .. | 1·78 | 1·60 | 1·40 | 2·50 | 2·81 | 2·21 | 1·98 |
| Water .. .. .. .. .. .. .. .... | 33·66 | 31·0 | 25·20 | 30·90 | 30·17 | 24·29 | 21·62 |
| Undetermined.. .. .. .. .. .. .. | 3·57 | 3·50 | 3·10 | — | 0·02 | — | — |
| Invert Sugar: Ash .. .. .. .. .... | 2·59 | 11·60 | 22·40 | 0·16 | 1·7 | 20·9 | 30·2 |
| Sucrose on 100 water .. .. .. .. .. | 168 | 146 | 145 | 214 | 207 | 112 ? | 77 ? |

| Constituents. | Potassium Sulphate. | | | Calcium Chloride. | | | Sulphate and Acetate of Potassium. | | |
|---|---|---|---|---|---|---|---|---|---|
| | 26 | 27 | 28 | 29 | 30 | 31 | 32 | 33 | 34 |
| *Weight in grammes:* | | | | | | | | | |
| Sucrose crystallized out .. | 2 | — | — | 30·3 | — | — | 27·0 | 38·1 | 38 7 |
| Sucrose dissolved .. .... | 103·5 | 102 | 58·5 | 91·5 | 95·0 | 70·5 | 93·5 | 82·5 | 79·5 |
| Sucrose inverted.. .. .. | 14·5 | 18·0 | 61·5 | — | 25·0 | 49·5 | — | — | 2·8 |
| *Composition of the solution in per cent.:—* | | | | | | | | | |
| Sucrose.. .. .. .. .. | 58·10 | 49·70 | 24·70 | 60·50 | 47·40 | 30·0 | 59·50 | 48·20 | 40·10 |
| Invert sugar .. .. .... | 11·65 | 24·0 | 52·34 | 4·60 | 25·60 | 47·20 | 4·50 | 18·0 | 30·01 |
| Ash .. .. .. .. .. | 2·13 | 1·75 | 1·72 | 1·80 | 1·40 | 1·22 | 3·76 | 3·24 | 3·78 |
| Water .. .. .. .... | 28·12 | 24·42 | 21·11 | 33·10 | 25·10 | 21·30 | 31·25 | 29·20 | 25·12 |
| Undetermined .. .. .. | — | 0·13 | 0·13 | — | 0·5 | 0·28 | 0·99 | 1·36 | 0·91 |
| Invert sugar: Ash.. .... | 2·7 | 13·7 | 30·5 | 2·5 | 18·0 | 37·9 | 1·2 | 5·5 | 8·0 |
| Sucrose on 100 water .... | 207 | 204 ? | 117 ? | 183 | 190 ? | 141 ? | 187 | 165 | 159 |

| Constituents. | Sodium Chloride and Acetate. | | | | Calcium Chloride and Lime Acetate. | | |
|---|---|---|---|---|---|---|---|
| | 35 | 36 | 37 | 38 | 39 | 40 | 41 |
| *Weight in grammes :—* | | | | | | | |
| Sucrose crystallized out .. .. .. .. | 10·7 | 17·2 | 28·2 | 29·7 | 25·1 | 42·1 | 50·3 |
| Sucrose dissolved .. .. .. .. .... | 109·0 | 102·5 | 90·0 | 87·5 | 94·5 | 76·0 | 68·5 |
| Sucrose inverted .. .. .. .. .. .. | — | — | 1·8 | 2·8 | — | — | 1·2 |
| *Composition of the solution in per cent.:—* | | | | | | | |
| Sucrose .. .. .. .. .. .. .. .. | 63·60 | 61·60 | 51·30 | 43·80 | 59·40 | 46·30 | 36·80 |
| Invert sugar .. .. .. .. .. .... | 0·70 | 4·20 | 16·10 | 28·50 | 4·01 | 18·31 | 32·10 |
| Ash .. .. .. .. .. .. .. .. .. | 5·50 | 3·60 | 3·33 | 2·76 | 4·05 | 3·60 | 2·97 |
| Water .. .. .. .. .. .. .. .... | 29·20 | 30·0 | 28·57 | 24·39 | 32·05 | 30·49 | 26·79 |
| Undetermined.. .. .. .. .. .. .. | 1·0 | 0·60 | 0·70 | 0·55 | 0·49 | 1·30 | 1·34 |
| Invert sugar : Ash .. .. .. .. .... | 0·13 | 1·16 | 4·8 | 10·3 | 1·0 | 5·0 | 10·8 |
| Sucrose on 100 water .. .. .. .. .. | 218 | 205 | 180 | 175 | 189 | 152 | 137 |

The mixtures containing glucose and inorganic salts revealed a striking fact; their joint presence gave rise to so much free acid from the inorganic salt, that at the high temperature at which the solutions were made some sucrose was inverted.

**Inversion by simultaneous presence of Invert Sugar and Inorganic Salt.**

For this reason the solutions Nos. 24, 25, 27, 28, 30, and 31 must be excluded from our calculations, as in these so much sucrose was inverted that there was not sufficient left to form a saturated solution. Not only had no sucrose crystallized out, but even the 2 grms. of sugar, used to start the crystallization, were dissolved in every case. The action of inorganic salts on the solubility of sucrose in presence of glucose could not therefore be studied, while

their influence in the absence of that body is sufficiently known from Herzfeld's experiments. When organic salts were also present in solution, the inversion was much less, and in every case a fully saturated solution resulted, as is shown by the large quantities of sucrose which crystallized. Owing to this circumstance, it was only possible to study the action of inorganic salts by comparing them with mixtures containing organic ones.

We observe that in every case, where glucose and salts are present in the solutions, the amount of sucrose crystallizing out increases in proportion as the liquid contains more glucose for the same amount of salts.

**Identical action of Organic and Inorganic Salts.**

In the mixtures containing both organic and inorganic salts, this crystallization is analogous with that found in those which contain the same amount of organic salts only, which shows that in this respect the action of organic and inorganic salts does not differ. So the mixtures Nos. 33, 34, 37, 38, 40, and 41 are respectively the same as Nos. 5, 6, 8, 9, 11, and 12, only that a part of the acetate has been replaced by an equivalent amount of an inorganic salt, and yet the weights of sucrose crystallized out in every set of two mixtures are in the same direction. A completely identical result cannot be expected as every salt has an action of its own, but we see that substitution of a part of the organic salt by an equivalent weight of an inorganic one has a slight tendency to promote crystallization of the sucrose.

| No. of the Mixture. | Salts. | Grms. of Invert Sugar. | Grms. of Sucrose. Crystallized out. | Dissolved. | Inverted. |
|---|---|---|---|---|---|
| 5 | Potassium acetate | 30 | 24·3 | 96·0 | 4·7 |
| 33 | Acetate and sulphate of potassium | 30 | 38·1 | 82·5 | — |
| 6 | Potassium acetate | 60 | 39·0 | 80·5 | — |
| 34 | Acetate and sulphate of potassium | 60 | 38·7 | 79·5 | 2·8 |
| 8 | Sodium acetate | 30 | 14·3 | 104·0 | 1·7 |
| 37 | Sodium acetate and sodium chloride | 30 | 29·7 | 90·0 | 1·8 |
| 9 | Sodium acetate | 60 | 18·2 | 97·0 | 4·8 |
| 38 | Sodium acetate and sodium chloride | 60 | 29·7 | 87·5 | 2·8 |
| 11 | Calcium acetate | 30 | 36·8 | 81·0 | 2·2 |
| 40 | Calcium acetate and calcium chloride | 30 | 42·1 | 76·0 | — |
| 12 | Calcium acetate | 60 | 40·1 | 79·0 | — |
| 41 | Calcium acetate and calcium chloride | 60 | 50·3 | 68·5 | 1·2 |

The calcium salts exert the strongest influence, next come potash salts, and finally soda salts.

Having seen that the simultaneous presence of any kind of salt and glucose promotes crystallization of sucrose, and that crystallization is more abundant when, for a given amount of salt, the proportion of glucose is greatest, we can take a further step and explain the whole theory of molasses formation.

We have already remarked that in the absence of reducing sugar, molasses is a hydrated combination between salts and sucrose, which is stable in a concentrated state and is only dissociated in a very dilute state. If we add invert sugar to that combination, the salts will combine with the glucose and fructose just as they did with sucrose, giving rise to a sucrose-glucose-fructose-salt combination, or more shortly, a sugar-salt combination. The proportion in which the five components (three sugars, water and salts) occurs depends on the nature of the salt; but in presence of glucose or invert sugar, less sucrose enters into combination for the same amount of salt, than when invert sugar is not present in the mixture. This amount of sucrose then becomes free, and can crystallize out.

**Invert Sugar can take the place of Sucrose in the Combinations.**

If this hypothesis be true, then, when mixing dry glucose with a quantity of the sucrose-salt combination, we must expect to see a part of the sucrose displaced by the invert sugar, and so enabled to crystallize.

For the same reason, when bringing together a saturated solution of sucrose with glucose and a dry salt, the salt will combine with the sugars and with water, and if the conditions of the experiment are well chosen, there will not remain sufficient free water left to keep all of the still free sucrose in solution; that surplus will therefore crystallize.

**Experiments proving this Substitution.**

With a view to proving these facts experimentally, the undermentioned mixtures were kept until all the supersaturated sugar had crystallized.

| | | | | | | |
|---|---|---|---|---|---|---|
| I. | 120 gr. | sucrose, | 50 gr. | water, | 10 gr. | potassium acetate. |
| II. | 120 | ,, | 50 | ,, | | |
| III. | 120 | ,, | 50 | ,, | 100 gr. | anhydrous glucose. |

As was to be expected from the foregoing experiments, Nos. II. and III. deposited about 15 grms. of sucrose, while the quantity of 10 grms. of acetate of potash added to No. I. was so chosen because that proportion causes 15 grms. of sucrose to crystallize, leaving about the same ratio of sucrose to water in the fully saturated mother-liquor. These clear syrupy fluids were transferred to other flasks, and mixed with 100 grms. of anhydrous glucose in either No. I. or II., and with 10 grms. of dry acetate of potash in No. III., after which the flasks were again set aside for crystallization. After six weeks 21 grms. of sucrose had crystallized in No. I., nothing in No. II., and 24 grms. in No. III.; which is, therefore, in full agreement with the theory. By way of confirmation, however, the experiment was repeated by mixing 300 grms. of a saturated solution of sucrose and acetate of potash with 225 grms. of anhydrous glucose. Another portion of that solution was kept without any addition as a check, and both were put aside for a couple of months. The former solution (containing glucose) yielded 75 grms. of sucrose, while the check solution (without addition) remained wholly uncrystallized.

The composition of the mother-liquor and the solution was as follows:—

| | Original. | With Glucose. |
|---|---|---|
| Sucrose | 64·4 | 37·5 |
| Glucose | 2·94 | 36·6 |
| Water | 30·80 | 24 |
| Ash | 0·97 | 1·26 |
| Undetermined | 0·89 | 0·64 |
| | 100 | 100 |

On 100 parts of syrup we added 45 grms. of glucose whilst 15 grms. of sucrose crystallized out, bringing the weight of the second syrup to 130 grms. When calculating the probable composition of the mother-liquor we get these values:—

$$\text{Sucrose } \frac{(64{\cdot}4 - 15) \times 100}{130} = 38 \text{ (found to be } 37{\cdot}5).$$

$$\text{Glucose } \frac{(45 + 2{\cdot}94) \times 100}{130} = 36{\cdot}8 \text{ (found to be } 36{\cdot}6).$$

$$\text{Water } \frac{30{\cdot}8 \times 100}{130} = 23{\cdot}7 \text{ (found to be } 24).$$

**Definition of an Ideal Cane Molasses.**

There is thus full agreement between the calculated composition and the figures found on analysis, which clearly proves that in a glucose-free molasses any addition of glucose can displace a part of the sucrose from the combination and cause it to crystallize. This brings us to the ultimate conclusion and definition of molasses, viz.: *Molasses is a hydrated combination between sugar and salts, which cannot be broken up by dissociation in a concentrated state, and therefore cannot yield sucrose in a crystallized form.*

**Its Composition.**

The total amount of sugars (sucrose + glucose + fructose) in an exhausted cane molasses is about 55 per cent.; and their relative proportions depend solely on the ratio of reducing sugars (glucose + fructose) to ash. The water content is about 20 per cent., and the ash about 9 per cent. In the case of juice containing no reducing sugar, the salts combine with sucrose only, and sucrose is the only sugar represented in the 55 per cent., so that the proportion between sucrose and water is in this case as 55 : 20. If, on the contrary, the juice contains reducing sugar as well, then this will also combine with a part of the salts, causing the total salts to hold less sucrose in combination. If, for instance, the amount of reducing sugar be 10 per cent., and of sucrose 45 per cent., then on 20 parts of water we find only 45 parts of sucrose; and in a case (which often occurs in cane molasses) of 30 sucrose and 25 reducing sugar, we only find a proportion of 30 sucrose to 20 water.

**Relation between Sucrose and Water in Presence of Invert Sugar.**

The supposed increase or decrease of the solubility of sucrose in the water of the molasses has long dominated the study of molasses-formation. If we accept that idea we notice the following solubilities for the corresponding relations between sucrose and invert sugar: —

| | | | | | |
|---|---|---|---|---|---|
| Invert Sugar | 0 | .... | 275 | parts of sucrose on | 100 water. |
| ,, | ,, 5 | .... | 250 | ,, ,, | ,, |
| ,, | ,, 10 | .... | 225 | ,, ,, | ,, |
| ,, | ,, 15 | .... | 200 | ,, ,, | ,, |
| ,, | ,, 20 | .... | 175 | ,, ,, | ,, |
| ,, | ,, 25 | .... | 150 | ,, ,, | ,, |

We see that the so called solubility of sucrose falls when the amount of glucose (or, in general, invert or reducing sugar) rises. Generally speaking, we can say that with a high ratio between glucose and ash, the solubility of sucrose decreases.

But this statement is far from being precise, and is apt to give rise to much confusion. There is no question of solubility of sucrose at all, but only of the composition of a combination, which contains on an average 55 per cent. sugars, 25 per cent. salts, and 20 per cent. water. If there is a large proportion of reducing sugar in that 55 per cent., then there is little sucrose; if, on the contrary, the percentage of glucose is small, the syrupy combination contains much sucrose. The ratio between the sucrose and water in such combinations has nothing whatever to do with a positive or negative melassigenic power of the constituents of the molasses.

It is evident that in the author's theory there is no place for negative molasses-formers; the non-sugars, especially salts, combine with sugars, and it depends entirely on the mutual relation of sucrose and glucose whether much or little sucrose enters into combination.

Further, it is clear that the conditions of the formation of molasses must be studied under similar circumstances as occur in practical working. We must, therefore, start from an excess of sugars (chiefly sucrose) over salts, because in practical sugar manufacture the sucrose always predominates in the juices and syrups; it constantly grows less through consecutive crystallizations, until no more is left than just corresponds to the sucrose content of the combinations mentioned.

Hence, in this work, the solubility of sucrose is not studied beyond the stage where the juice is concentrated to syrup. Free sucrose still preponderates in the concentrated syrups, as compared with the small quantity of combined sucrose; but in the first molasses (from which a great deal of sucrose has

been withdrawn) this proportion is already so disturbed that it may be regarded as a solution of sucrose in a hydrated compound of sugar and salts. Finally, the exhausted molasses consists solely of this hydrated compound, wherein the ratio of sucrose to water bears no relation to the solubility of sucrose in an uncombined state.

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## II.—Composition of Exhausted Molasses.

The composition of exhausted Java molasses appears in the tables which follow, and differs very little from that of exhausted molasses produced in other countries.

The molasses are divided into two groups, viz., those obtained from the final mixed masse cuites boiled to grain (see page 227) and those obtained from masse cuites boiled to string-proof and cooled in tanks.

A subdivision is made according to the method of clarification, whether by defecation or by carbonatation.

**Molasses may be Diluted or may contain Crystals in Suspension.**

As the molasses were obtained in the usual course of manufacture they are probably somewhat diluted, or may contain minute sugar crystals in suspension, and are therefore not identical with the mother-liquor from the final crystallization.

Referring to the analyses which follow, numbers 30, 48, 49, 66, and 77, are so liquid that they must have been subsequently diluted, and Nos. 49 and 77 to such an extent that a part of the sucrose has become inverted by fermentation, so that the sucrose, quotient of purity, and other figures derived therefrom, could not be determined, but only the figures for ash, gummy matter, and the composition of the ash.

Nos. 4, 6, 7, 8, 10, 13, 15, 16, 17, 18, 20, 21, 27, 33, 35, 38, 44, 50, 52, 55, 60, 63, 65, 68, 69, 70, 71, 72, and 75, contained a small quantity of suspended crystals, but too small in quantity to be determined. The presence of crystals affords proof that the molasses are fully saturated, otherwise sucrose could not have crystallized out.

The analyses were made by the methods described in the *International Sugar Journal*, 1901, 519, and 1906, 27.

## I.—Molasses from Masse Cuites, Boiled to Grain.

| Number. | Method of Clarification. | Purity. Original Juice. | Degrees Brix. | Actual Dry Substance. | Direct Polarization. | Polarization after Clerget. | Apparent Purity. | Real Purity. | Reducing Sugar. | Ash. | Gummy Matter. | Organic Non-Sugar. | Rotatory power of the Reducing Sugar. | Factor for the difference between Brix and dry substance. | Sucrose dissolved in 100 parts of water. | Ratio between Reducing Sugar and Ash. |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | Carb. | — | 84·56 | 80·43 | 32·6 | 34·42 | 38·55 | 42·79 | 22·18 | 7·61 | 0·86 | 16·22 | — 8·2 | 0·53 | 125 | 2·91 |
| 2 | ,, | 89·2 | 82·10 | 78·60 | 32·2 | 31·93 | 39·22 | 40·62 | 21·74 | 7·26 | 0·51 | 17·67 | + 1·3 | 0·48 | 149 | 3·00 |
| 3 | ,, | 86·5 | 81·84 | 77·60 | 28·6 | 28·15 | 34·95 | 36·15 | 25·00 | 7·45 | 0·59 | 17·00 | + 1·8 | 0·57 | 121 | 3·36 |
| 4 | ,, | — | 86·78 | 82·18 | 28·8 | 30·6 | 33·19 | 37·24 | 22·00 | 10·80 | 0·89 | 18·78 | — 8·2 | 0·47 | 172 | 2·25 |
| 5 | Def. | 82·37 | 95·61 | 88·04 | 33·8 | 40·65 | 35·52 | 46·17 | 21·74 | 12·94 | 2·39 | 12·71 | — 31·5 | 0·55 | 339 | 1·67 |
| 6 | ,, | — | 86·42 | 80·98 | 29·4 | 34·1 | 39·53 | 42·11 | 23·2 | 8·88 | 2·36 | 14·80 | — 20·3 | 0·60 | 179 | 2·62 |
| 7 | ,, | 82·43 | 87·39 | 82·48 | 32·8 | 36·52 | 37·53 | 44·28 | 23·25 | 8·02 | 1·72 | 10·59 | — 16·0 | 0·61 | 209 | 2·90 |
| 8 | ,, | 90·3 | 85·64 | 77·64 | 30·4 | 37·65 | 35·56 | 48·50 | 17·24 | 9·23 | 2·57 | 13·52 | — 12·1 | 0·87 | 168 | 1·87 |
| 9 | ,, | 82·0 | 87·02 | 81·30 | 32·6 | 38·43 | 36·32 | 47·27 | 21·73 | 8·21 | 1·71 | 12·83 | — 26·8 | 0·68 | 206 | 2·65 |
| 10 | ,, | 85·1 | 91·92 | 85·41 | 31·1 | 35·8 | 33·83 | 41·92 | 24·6 | 10·45 | 1·22 | 14·56 | — 19·1 | 0·62 | 244 | 2·35 |
| 11 | ,, | 84·06 | 88·32 | 81·76 | 31·8 | 36·05 | 36·00 | 45·35 | 23·81 | 9·23 | 1·84 | 12·67 | — 19·6 | 0 71 | 197 | 2·58 |
| 12 | ,, | 85·3 | 92·56 | 87·70 | 33·2 | 39·0 | 35·86 | 44·47 | 29·4 | 8·00 | 2·01 | 11·23 | — 19·7 | 0·61 | 317 | 3·67 |
| 13 | ,, | 86·9 | 88·42 | 81·15 | 32·2 | 37·80 | 37·26 | 46·58 | 22·73 | 8·39 | 2·00 | 12·23 | — 24·7 | 0·63 | 205 | 2·71 |
| 14 | ,, | 81·4 | 87·14 | 82·26 | 27·4 | 32·49 | 31·44 | 39·50 | 29·41 | 7·70 | 1·09 | 12·66 | — 17·3 | 0·63 | 183 | 3·82 |
| 15 | ,, | 84·4 | 88·48 | 82·96 | 29·8 | 36·1 | 33·69 | 43·51 | 22·20 | 7·39 | 2·00 | 17·27 | — 28·4 | 0·75 | 208 | 3·00 |
| 16 | ,, | 87·5 | 87·26 | 82·85 | 22·8 | 29·96 | 26·13 | 36·16 | 25·64 | 7·96 | 2·27 | 19·29 | — 27·9 | 0·56 | 175 | 3·22 |
| 17 | ,, | 85·8 | 89·37 | 83·94 | 39·2 | 42·44 | 43·86 | 50·56 | 21·28 | 9·06 | 1·51 | 11·16 | — 15·2 | 0·60 | 265 | 2·35 |
| 18 | ,, | 86·3 | 85·36 | 80·92 | 27·0 | 32·29 | 31·51 | 38·94 | 29·41 | 6·32 | 2·29 | 13·65 | — 17·0 | 0·70 | 190 | 4·65 |
| 19 | ,, | — | 79·98 | 73·08 | 26·1 | 29·37 | 32·50 | 40·19 | 20·00 | 8·37 | 2·62 | 15·34 | — 16·3 | 0·82 | 109 | 2·44 |
| 20 | ,, | 85·65 | 90·60 | 85·10 | 29·3 | 36·3 | 32·34 | 42·64 | 22·8 | 8·75 | 2·13 | 17·25 | — 30·7 | 0·63 | 244 | 2·61 |
| 21 | ,, | 83·2 | 87·38 | 83·61 | 26·0 | 32·31 | 29·75 | 38·64 | 27·03 | 7·61 | 1·72 | 15·66 | — 34·5 | 0·50 | 197 | 3·55 |
| 22 | ,, | 86·22 | 80·64 | 76·42 | 33·2 | 36·9 | 41·17 | 48·29 | 21·3 | 7·33 | 1·50 | 10·89 | — 17·4 | 0·58 | 152 | 2·91 |

| Number. | Method of Clarification. | Difference in Purity (apparent) between Juice and Molasses. | Difference in Purity (real) between Juice and Molasses. | Total Ash. | Ash insoluble in water. | Ash soluble in water. | Iron and Calcium Phosphate. | Lime. | Magnesia. | Potash. | Soda. | Silica. | Sulphuric Acid. | Carbonic Acid in the Ash. | Chlorine. | Lime in 100 parts of Ash. |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | Carb. | — | — | 7·61 | 1·13 | 6·48 | 0·12 | 0·48 | 0·08 | 3·86 | 0·08 | 0·03 | 0·75 | 1·69 | 0·43 | 6·31 |
| 2 | ,, | 49·98 | 48·58 | 7·26 | 2·13 | 5·13 | 0·12 | 1·16 | 0·02 | 3·38 | 0·13 | 0·02 | 0·23 | 2·19 | 0·16 | 16·01 |
| 3 | ,, | 51·55 | 50·35 | 7·45 | 2·32 | 5·13 | 0·15 | 1·24 | 0·04 | 3·27 | 0·15 | 0·03 | 0·29 | 2·22 | 0·16 | 6·40 |
| 4 | ,, | — | — | 9·47 | 1·39 | 8·08 | 0·15 | 0·68 | 0·02 | 5·11 | 0·00 | 0·15 | 0·74 | 2·56 | 0·37 | 7·19 |
| 5 | Def. | 46·85 | 36·20 | 12·94 | 1·67 | 11·27 | 0·76 | 0·25 | 0·29 | 6·47 | 0·00 | 0·21 | 1·61 | 1·09 | 2·52 | 1·93 |
| 6 | ,, | — | — | 8·88 | 2·04 | 6·84 | 0·62 | 0·52 | 0·17 | 3·96 | 0·00 | 0·27 | 1·18 | 1·39 | 0·52 | 5·85 |
| 7 | ,, | 44·90 | 38·15 | 8·02 | 1·88 | 6·14 | 0·49 | 0·67 | 0·20 | 3·61 | 0·02 | 0·18 | 1·26 | 1·33 | 0·42 | 8·35 |
| 8 | ,, | 54·74 | 41·80 | 9·23 | 1·87 | 7·36 | 0·82 | 0·57 | 0·07 | 4·25 | 0·57 | 0·15 | 1·14 | 1·61 | 0·38 | 6·17 |
| 9 | ,, | 45·68 | 34·73 | 8·21 | 1·66 | 6·55 | 0·42 | 0·96 | 0·16 | 3·93 | 0·20 | 0·16 | 0·82 | 1·69 | 0·34 | 11·69 |
| 10 | ,, | 51·27 | 43·18 | 10·45 | 1·75 | 8·70 | 0·44 | 0·37 | 0·15 | 5·48 | 0·12 | 0·22 | 0·69 | 2·27 | 0·99 | 3·54 |
| 11 | ,, | 48·06 | 38·71 | 9·23 | 2·18 | 7·05 | 0·41 | 0·85 | 0·17 | 4·01 | 0·04 | 0·15 | 1·44 | 1·60 | 0·64 | 9·21 |
| 12 | ,, | 49·44 | 40·83 | 8·00 | 2·75 | 5·25 | 0·52 | 0·67 | 0·10 | 3·34 | 0·00 | 0·51 | 0·86 | 1·46 | 0·17 | 8·37 |
| 13 | ,, | 49·64 | 40·32 | 8·39 | 2·10 | 6·29 | 0·76 | 0·47 | 0·49 | 3·76 | 0·13 | 0·34 | 1·21 | 1·15 | 0·68 | 5·61 |
| 14 | ,, | 49·96 | 41·90 | 7·70 | 1·55 | 6·15 | 0·38 | 0·52 | 0·26 | 3·62 | 0·00 | 0·10 | 1·08 | 1·35 | 0·28 | 6·76 |
| 15 | ,, | 50·71 | 39·89 | 7·39 | 2·99 | 5·40 | 0·76 | 0·45 | 0·19 | 3·31 | 0·06 | 0·42 | 0·67 | 1·54 | 0·25 | 6·09 |
| 16 | ,, | 61·37 | 51·34 | 7·96 | 2·05 | 5·91 | 0·92 | 0·50 | 0·08 | 3·32 | 0·07 | 0·17 | 1·24 | 1·19 | 0·37 | 6·28 |
| 17 | ,, | 41·94 | 35·24 | 9·06 | 1·99 | 7·07 | 0·34 | 0·69 | 0·13 | 4·03 | 0·02 | 0·21 | 1·37 | 1·19 | 0·85 | 7·62 |
| 18 | ,, | 54·79 | 37·36 | 6·32 | 2·00 | 4·32 | 0·89 | 0·48 | 0·05 | 2·62 | 0·07 | 0·23 | 0·85 | 1·03 | 0·68 | 7·59 |
| 19 | ,, | — | — | 8·37 | 1·88 | 6·54 | 1·13 | 0·29 | 0·05 | 3·89 | 0·11 | 0·11 | 0·91 | 1·67 | 0·54 | 3·47 |
| 20 | ,, | 53·31 | 43·00 | 8·75 | 1·86 | 6·89 | 0·83 | 0·31 | 0·16 | 4·32 | 0·13 | 0·20 | 0·56 | 1·91 | 0·38 | 9·49 |
| 21 | ,, | 53·54 | 44·56 | 7·61 | 1·44 | 6·17 | 0·63 | 0·06 | 0·13 | 4·20 | 0·06 | 0·18 | 0·87 | 1·18 | 0·34 | 8·28 |
| 22 | ,, | 45·05 | 37·93 | 7·33 | 2·39 | 4·94 | 0·61 | 0·49 | 0·14 | 2·98 | 0·06 | 0·35 | 0·85 | 1·19 | 0·35 | 6·68 |

## II.—Molasses from Masse Cuites Boiled to String-proof and Cooled in Tanks.

| Number. | Method of Clarification. | Purity. Original Juice. | Degrees Brix. | Actual Dry Substance. | Direct Polarization. | Polarization after Clerget. | Apparent Purity. | Real Purity. | Reducing Sugar. | Ash. | Gummy Matter. | Organic Non-Sugar. | Rotatory Power of the Reducing Sugar. | Factor for the difference between Brix and Dry Substance. | Sucrose dissolved in 100 parts of water. | Ratio between Reducing Sugar and Ash. |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 23 | Carb. | 85·00 | 82·98 | 77·80 | 30·3 | 29 9 | 36·63 | 38·43 | 26 3 | 8·49 | 0·83 | 13·11 | + 1·3 | 0·61 | 135 | 3·09 |
| 24 | ,, | 81·5 | 83·38 | 78·49 | 31·6 | 32·13 | 37·90 | 40·94 | 19·61 | 9·21 | 0·85 | 17·54 | — 2·6 | 0·52 | 150 | 2·13 |
| 25 | ,, | 82·7 | 83·18 | 76·66 | 33·8 | 34·1 | 40·63 | 44·48 | 15·4 | 9·04 | 0·61 | 18·12 | — 2·0 | 0·72 | 147 | 1·70 |
| 26 | ,, | — | 84·82 | 77·64 | 30·8 | 30·9 | 36·31 | 39·94 | 21·7 | 8·03 | 0·70 | 17·10 | — 0·5 | 0·89 | 134 | 2·70 |
| 27 | Def. | 85·24 | 81·94 | 76·72 | 30·2 | 33·6 | 36·86 | 43·80 | 21·2 | 8·60 | 1·11 | 13·32 | — 16·0 | 0·61 | 145 | 2·47 |
| 28 | ,, | 86·3 | 84·66 | 77·36 | 27·6 | 33·78 | 32·60 | 43·67 | 24·39 | 9·24 | 2·47 | 9·95 | — 27·8 | 0·79 | 149 | 2·64 |
| 29 | ,, | 83·44 | 80·92 | 77·52 | 23·8 | 27·7 | 29·41 | 35·86 | 27·8 | 7·14 | 1·69 | 14·88 | — 14·0 | 0·48 | 123 | 3·89 |
| 30 | ,, | 88·9 | 72·96 | 69·96 | 27·0 | 27·78 | 37·01 | 39·69 | 16·95 | 7·22 | 1·60 | 18·01 | — 4·5 | 0·42 | 92 | 2·35 |
| 31 | ,, | 89·94 | 87·00 | 82·76 | 29·8 | 32·1 | 34·25 | 38·79 | 26·3 | 8·09 | 1·91 | 16·27 | — 8·7 | 0·52 | 186 | 3·25 |
| 32 | ,, | 84·57 | 77·18 | 73·52 | 27·6 | 29·2 | 35·76 | 39·72 | 27·8 | 7·43 | 1·34 | 9·09 | — 5·8 | 0·49 | 111 | 3·61 |
| 33 | ,, | 89·27 | 89·18 | 84·50 | 35·3 | 40·6 | 39·58 | 48·02 | 22·8 | 8·35 | 1·44 | 12·45 | — 24·5 | 0·56 | 264 | 2·73 |
| 34 | ,, | 89·12 | 83·76 | 77·92 | 30·4 | 33·5 | 36·29 | 43·00 | 17·51 | 9·20 | 1·54 | 17·71 | — 17·7 | 0·63 | 152 | 1·90 |
| 35 | ,, | — | 85·04 | 78·40 | 24·9 | 27·9 | 29·29 | 35·59 | 27·8 | 8 32 | 1·89 | 14·38 | — 10·8 | 0·80 | 130 | 3·34 |
| 36 | ,, | 82·49 | 87·75 | 80·72 | 26·2 | 33·11 | 29·86 | 41·18 | 22·73 | 11·40 | 2·08 | 13·48 | — 30·4 | 0·62 | 172 | 1·98 |
| 37 | ,, | 83·77 | 87·12 | 81·53 | 26·8 | 30·76 | 33·85 | 36·17 | 29·94 | 9·31 | 1·66 | 12·79 | — 9·0 | 0·60 | 161 | 3·22 |
| 38 | ,, | — | 85·65 | 78·86 | 26·2 | 33·42 | 30·59 | 42·38 | 24·39 | 9·20 | 1·64 | 11·15 | — 29·6 | 0·76 | 158 | 2·65 |
| 39 | ,, | 86·3 | 82·98 | 79·32 | 28·6 | 31·99 | 34·47 | 40·33 | 27·03 | 7·04 | 1·50 | 13·26 | — 12·5 | 0·52 | 155 | 3·98 |
| 40 | ,, | 90·5 | 84·08 | 77·32 | 32·6 | 34·44 | 38·77 | 44·54 | 16·13 | 11·10 | 1·90 | 15·65 | — 11·4 | 0·66 | 150 | 1·45 |
| 41 | ,, | 90·33 | 79·28 | 74·98 | 28·6 | 32·02 | 36·07 | 42·68 | 20·00 | 7·52 | 2·27 | 15·41 | — 17·1 | 0·57 | 128 | 2·66 |

| Number. | Method of Clarification. | Difference in Purity (apparent) between Juice and Molasses. | Difference in Purity (real) between Juice and Molasses. | Total Ash. | Ash insoluble in Water. | Ash soluble in Water. | Iron and Calcium Phosphate. | Lime. | Magnesia. | Potash. | Soda. | Silica. | Sulphuric Acid. | Carbonic Acid in the Ash. | Chlorine. | Lime in 100 parts of Ash. |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 23 | Carb. | 48·37 | 46·57 | 8·49 | 2·94 | 5·55 | 0·15 | 1·62 | 0·02 | 3·44 | 0·15 | 0·04 | 0·40 | 2·17 | 0·68 | 19·08 |
| 24 | ,, | 43·6 | 40·56 | 9·21 | 3·89 | 5·32 | 0·19 | 2·08 | 0·05 | 2·48 | 0·10 | 0·03 | 0·40 | 3·83 | 0·61 | 21·49 |
| 25 | ,, | 42·07 | 38·22 | 9·01 | 3·04 | 6·00 | 0·09 | 1·62 | 0·00 | 3·99 | 0·17 | 0·00 | 0·16 | 2·70 | 0·37 | 17·92 |
| 26 | ,, | — | — | 8·03 | 4·18 | 3·85 | 0·16 | 2·18 | 0·00 | 2·32 | 0·00 | 0·03 | 0·49 | 2·48 | 0·21 | 20·17 |
| 27 | Def. | 48·38 | 41·44 | 8·60 | 2·40 | 6·20 | 0·37 | 0·77 | 0·14 | 3·76 | 0·44 | 0·10 | 1·01 | 1·32 | 0·45 | 8·95 |
| 28 | ,, | 53·7 | 42·63 | 9·24 | 1·53 | 7·71 | 0·91 | 0·20 | 0·07 | 4·68 | 0·15 | 0·18 | 0·85 | 1·49 | 0·72 | 2·16 |
| 29 | ,, | 54·03 | 47·58 | 7·14 | 1·93 | 5·21 | 0·65 | 0·48 | 0·09 | 3·17 | 0·09 | 0·22 | 0·65 | 1·43 | 0·20 | 6·72 |
| 30 | ,, | 51·89 | 49·21 | 7·22 | 1·84 | 5·38 | 0·55 | 0·54 | 0·15 | 3·24 | 0·06 | 0·16 | 0·74 | 1·50 | 0·24 | 7·48 |
| 31 | ,, | 55·69 | 51·15 | 8·09 | 2·43 | 5·66 | 1·07 | 0·53 | 0·10 | 3·54 | 0·00 | 0·30 | 0·30 | 1·95 | 0·27 | 6·55 |
| 32 | ,, | 48·81 | 44·85 | 7·43 | 1·85 | 5·58 | 0·60 | 0·46 | 0·09 | 3·31 | 0·14 | 0·30 | 0·86 | 1·17 | 0·58 | 6·19 |
| 33 | ,, | 49·69 | 41·25 | 8·35 | 2·06 | 6·29 | 0·75 | 0·30 | 0·11 | 3·90 | 0·08 | 0·28 | 0·85 | 1·35 | 0·40 | 3·59 |
| 34 | ,, | 52·83 | 46·12 | 9·20 | 2·87 | 6·33 | 0·42 | 1·25 | 0·08 | 3·96 | 0·00 | 0·06 | 0·87 | 2·22 | 0·21 | 13·59 |
| 35 | ,, | — | — | 8·32 | 2·51 | 5·81 | 0·66 | 0·65 | 0·26 | 3·44 | 0·25 | 0·36 | 1·19 | 1·54 | 0·23 | 7·93 |
| 36 | ,, | 52·63 | 41·31 | 11·40 | 1·55 | 9·85 | 0·61 | 0·33 | 0·20 | 6·03 | 0·06 | 0·17 | 0·84 | 1·77 | 0·84 | 2·90 |
| 37 | ,, | 53·01 | 47·60 | 9·31 | 2·37 | 6·94 | 0·51 | 0·80 | 0·05 | 4·07 | 0·09 | 0·24 | 1·37 | 1·49 | 0·71 | 8·59 |
| 38 | ,, | — | — | 9·20 | 1·17 | 8·03 | 0·52 | 0·24 | 0·10 | 5·04 | 0·10 | 0·11 | 0·48 | 2·05 | 0·51 | 2·61 |
| 39 | ,, | 51·8 | 45·97 | 7·04 | 1·74 | 5·30 | 0·54 | 0·60 | 0·08 | 3·08 | 0·10 | 0·06 | 0·82 | 1·44 | 0·20 | 8·52 |
| 40 | ,, | 51·73 | 45·96 | 11·10 | 2·10 | 9·00 | 0·55 | 0·73 | 0·22 | 5·35 | 0·06 | 0·03 | 1·29 | 1·91 | 0·95 | 6·58 |
| 41 | ,, | 58·83 | 47·55 | 7·52 | 2·33 | 5·19 | 0·94 | 0·46 | 0·20 | 3·10 | 0·04 | 0·31 | 0·86 | 1·39 | 0·21 | 6·12 |

## II.—Molasses from Masse Cuites Boiled to String-proof and Cooled in Tanks.—*Continued.*

| Number. | Method of Clarification. | Purity Original Juice. | Degrees Brix. | Actual Dry Substance. | Direct Polarization. | Polarization after Clerget. | Apparent Purity. | Real Purity. | Reducing Sugar. | Ash. | Gummy Matter. | Organic Non-Sugar. | Rotatory Power of the Reducing Sugar. | Factor for the difference between Brix and Dry Substance. | Sucrose dissolved in 100 parts of water. | Ratio between Reducing Sugar and Ash. |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 42 | Def. | — | 81·76 | 75·94 | 25·6 | 33·28 | 31·31 | 43·82 | 20·00 | 8·34 | 1·83 | 14·32 | — 33·4 | 0·69 | 138 | 2·40 |
| 43 | ,, | 83·62 | 84·58 | 78·50 | 27·4 | 34·00 | 32·39 | 43·31 | 20·00 | 9·58 | 2·02 | 14·92 | — 33·0 | 0·63 | 158 | 2·09 |
| 44 | ,, | — | 82·76 | 78·10 | 26·6 | 30·67 | 32·14 | 39·27 | 28·57 | 6·04 | 1·13 | 12·82 | — 14·3 | 0·77 | 140 | 4·73 |
| 45 | ,, | 86·82 | 84·44 | 80·44 | 21·8 | 26·1 | 25·82 | 32·45 | 32·3 | 7·97 | 1·22 | 14·07 | — 13·3 | 0·50 | 122 | 4·05 |
| 46 | ,, | 81·7 | 82·86 | 78·87 | 24·0 | 28·35 | 28·96 | 35·35 | 29·41 | 7·02 | 2·34 | 14·09 | — 14·8 | 0·56 | 134 | 4·17 |
| 47 | ,, | 89·5 | 79·18 | 74·06 | 31·8 | 34·75 | 40·02 | 46·92 | 16·13 | 8·40 | 3·97 | 14·78 | — 18·3 | 0·61 | 134 | 1·92 |
| 48 | ,, | 88·3 | 74·20 | 67·56 | 23·0 | 25·7 | 31·0 | 38·04 | 20·8 | 7·68 | 1·55 | 13·38 | — 13·0 | 0·86 | 80 | 2·71 |
| 49 | ,, | 84·90 | 68·65 | 66·30 | — | — | — | — | — | 6·24 | 1·22 | — | — | — | — | — |
| 50 | ,, | 89·0 | 91·16 | 85·30 | 31·8 | 37·20 | 34·88 | 43·61 | 21·28 | 9·98 | 2·62 | 16·84 | — 25·4 | 0·59 | 253 | 2·13 |
| 51 | ,, | 84·82 | 81·38 | 75·14 | 26·8 | 30·8 | 32·93 | 40·09 | 24·4 | 9·14 | 1·61 | 8·67 | — 16·4 | 0·68 | 124 | 2·67 |
| 52 | ,, | 82·7 | 87·30 | 78·83 | 25·4 | 32·28 | 29·10 | 40·95 | 25·64 | 9·62 | 2·44 | 11·29 | — 26·8 | 0·89 | 152 | 2·67 |
| 53 | ,, | 83·56 | 83·46 | 77·66 | 29·6 | 34·49 | 35·47 | 44·41 | 24·39 | 9·30 | 2·47 | 9·48 | — 20·5 | 0·62 | 154 | 2·62 |
| 54 | ,, | — | 86·14 | 80·32 | 25·8 | 31·39 | 29·95 | 39·08 | 27·03 | 8·31 | 1·61 | 13·59 | — 20·4 | 0·70 | 160 | 3·25 |
| 55 | ,, | 84·9 | 83·50 | 78·40 | 24·9 | 27·9 | 29·29 | 35·59 | 29·4 | 7·04 | 1·50 | 12·52 | — 17·4 | 0·77 | 133 | 4·17 |
| 56 | ,, | — | 83·12 | 77·42 | 27·4 | 32·91 | 32·93 | 42·52 | 27·03 | 7·92 | 1·39 | 9·56 | — 20·4 | 0·72 | 141 | 3·41 |
| 57 | ,, | 82·50 | 81·76 | 77·62 | 26·2 | 30·78 | 32·04 | 39·66 | 28·57 | 6·79 | 1·31 | 11·48 | — 16·0 | 0·61 | 138 | 4·21 |
| 58 | ,, | — | 81·80 | 74·46 | 27·4 | 32·72 | 33·49 | 43·94 | 20·41 | 9·00 | 4·41 | 12·33 | — 26·0 | 0·82 | 128 | 2·27 |
| 59 | ,, | — | 85·70 | 78·72 | 27·0 | 31·78 | 31·50 | 40·48 | 22·73 | 9·91 | 1·53 | 14·21 | — 21·4 | 0·70 | 150 | 2·40 |
| 60 | ,, | 85·47 | 84·40 | 80·06 | 29·2 | 32·12 | 34·60 | 40·12 | 22·73 | 8·77 | 3·14 | 16·44 | — 12·9 | 0·50 | 161 | 2·59 |

| Number. | Method of Clarification. | Difference in Purity (apparent) between Juice and Molasses. | Difference in Purity (real) between Juice and Molasses. | Total Ash. | Ash insoluble in Water. | Ash soluble in Water. | Iron and Calcium Phosphate. | Lime. | Magnesia. | Potash. | Soda. | Silica. | Sulphuric Acid. | Carbonic Acid in the Ash. | Chlorine. | Lime in 100 parts of Ash. |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 42 | Def. | — | — | 8·34 | 1·37 | 6·97 | 0·73 | 0·19 | 0·21 | 4·17 | 0·08 | 0·14 | 0·92 | 1·32 | 0·61 | 2·04 |
| 43 | ,, | 54·23 | 43·31 | 9·58 | 2·02 | 7·56 | 1·07 | 0·47 | 0·08 | 4·51 | 0·10 | 0·11 | 1·13 | 1·72 | 0·71 | 4·91 |
| 44 | ,, | — | — | 6·04 | 1·73 | 4·31 | 0·39 | 0·59 | 0·18 | 2·36 | 0·00 | 0·09 | 0·95 | 1·02 | 0·34 | 9·77 |
| 45 | ,, | 61·00 | 54·37 | 7·97 | 1·85 | 6·12 | 0·72 | 0·36 | 0·12 | 3·64 | 0·14 | 0·24 | 1·08 | 1·34 | 0·55 | 4·52 |
| 46 | ,, | 52·74 | 45·75 | 7·02 | 1·97 | 5·05 | 0·73 | 0·51 | 0·05 | 3·00 | 0·07 | 0·21 | 0·84 | 1·33 | 0·30 | 7·27 |
| 47 | ,, | 49·48 | 42·58 | 8·40 | 2·44 | 5·96 | 1·00 | 0·66 | 0·05 | 3·75 | 0·14 | 0·20 | 0·23 | 2·06 | 0·30 | 7·86 |
| 48 | ,, | 57·3 | 50·26 | 7·68 | 1·51 | 6·17 | 0·66 | 0·28 | 0·14 | 3·40 | 0·17 | 0·11 | 0·46 | 1·72 | 0·41 | 3·65 |
| 49 | ,, | — | — | 6·24 | 1·18 | 5·06 | 0·27 | 0·45 | 0·10 | 3·13 | 0·06 | 0·13 | 0·29 | 1·38 | 0·28 | 7·21 |
| 50 | ,, | 54·12 | 45·39 | 9·98 | 1·76 | 8·22 | 0·84 | 0·28 | 0·22 | 4·98 | 0·11 | 0·24 | 1·29 | 1·54 | 0·82 | 2·81 |
| 51 | ,, | 51·35 | 44·73 | 9·14 | 1·70 | 7·44 | 0·45 | 0·54 | 0·23 | 4·56 | 0·00 | 0·13 | 0·66 | 2·09 | 0·58 | 5·90 |
| 52 | ,, | 53·6 | 41·75 | 9·62 | 1·42 | 8·20 | 0·82 | 0·30 | 0·06 | 5·07 | 0·05 | 0·14 | 0·93 | 1·70 | 0·68 | 3·12 |
| 53 | ,, | 48·09 | 39·15 | 9·30 | 2·44 | 6·86 | 1·05 | 0·43 | 0·19 | 4·27 | 0·00 | 0·40 | 0·60 | 1·80 | 0·74 | 4·62 |
| 54 | ,, | — | — | 8·31 | 1·58 | 6·73 | 0·66 | 0·36 | 0·16 | 4·06 | 0·01 | 0·18 | 1·23 | 1·23 | 0·44 | 4·33 |
| 55 | ,, | 56·16 | 49·31 | 7·04 | 1·64 | 5·40 | 0·55 | 0·46 | 0·16 | 3·50 | 0·00 | 0·16 | 0·28 | 1·46 | 0·59 | 6·53 |
| 56 | ,, | — | — | 7·92 | 1·93 | 5·99 | 0·49 | 0·15 | 0·12 | 4·34 | 0·00 | 0·18 | 0·57 | 1·36 | 0·64 | 1·89 |
| 57 | ,, | 50·46 | 42·84 | 6·79 | 1·25 | 5·54 | 0·29 | 0·42 | 0·14 | 3·30 | 0·08 | 0·07 | 0·61 | 1·32 | 0·44 | 6·18 |
| 58 | ,, | — | — | 9·00 | 2·86 | 6·14 | 0·89 | 0·99 | 0·04 | 3·76 | 0·10 | 0·10 | 0·21 | 2·27 | 0·65 | 11·00 |
| 59 | ,, | — | — | 9·91 | 1·95 | 7·96 | 0·36 | 0·78 | 0·13 | 4·88 | 0·00 | 0·04 | 0·94 | 2·07 | 0·94 | 7·86 |
| 60 | ,, | 50·87 | 45·35 | 8·77 | 2·82 | 5·95 | 1·30 | 0·49 | 0·02 | 3·62 | 0·10 | 0·36 | 0·34 | 1·82 | 0·38 | 5·59 |

## II.—Molasses from Masse Cuites Boiled to String-proof and Cooled in Tanks.—*Continued.*

| Number. | Method of Clarification. | Purity Original Juice. | Degrees Brix. | Actual Dry Substance. | Direct Polarization. | Polarization after Clerget. | Apparent Purity. | Real Purity. | Reducing Sugar. | Ash. | Gummy Matter. | Organic Non-Sugar. | Rotatory Power of the Reducing Sugar. | Factor for the difference between Brix and Dry Substance. | Sucrose dissolved in 100 parts of water. | Ratio between Reducing Sugar and Ash. |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 61 | Def. | 82·1 | 82·42 | 78 54 | 29·0 | 31·3 | 35·19 | 46·91 | 27·8 | 7·89 | 1·14 | 8·55 | — 19·1 | 0·49 | 160 | 3 51 |
| 62 | ,, | 85·0 | 82·80 | 78·04 | 29·2 | 31·40 | 35·26 | 40 23 | 25·00 | 8 27 | 1·35 | 13·37 | — 8·8 | 0·57 | 144 | 3·02 |
| 63 | ,, | 88·7 | 85·30 | 78·84 | 27·4 | 32·81 | 32·12 | 41·62 | 19·61 | 10·16 | 2·33 | 16·36 | — 28·1 | 0·64 | 155 | 1·95 |
| 64 | ,, | 90·5 | 80·98 | 76·26 | 33·8 | 37·20 | 41·74 | 48·78 | 14·93 | 8·93 | 2·58 | 15·20 | — 22·9 | 0·5 | 159 | 1·66 |
| 65 | ,, | 85·0 | 88·74 | 84·10 | 32·6 | 35·42 | 36·73 | 42·12 | 26·32 | 9·55 | 1·49 | 12·81 | — 10·7 | 0·49 | 223 | 2·76 |
| 66 | ,, | — | 75·24 | 71·07 | 25·8 | 27·9 | 34·29 | 39·26 | 27·0 | 8·20 | 1·42 | 7·97 | — 7·8 | 0·51 | 96 | 3·29 |
| 67 | ,, | — | 79·70 | 76 12 | 28·1 | 34·09 | 35·25 | 44·78 | 22·22 | 6·90 | 3·92 | 13·91 | — 27·0 | 0·52 | 147 | 3·22 |
| 68 | ,, | 83·5 | 86·83 | 81·50 | 25·2 | 31·33 | 29·02 | 38·43 | 27·78 | 9·53 | 1·84 | 12·86 | — 22·7 | 0·56 | 170 | 2·92 |
| 69 | ,, | 84·0 | 83·10 | 77·41 | 28·8 | 35·79 | 34 66 | 46·22 | 22·22 | 8·41 | 1·72 | 11·02 | — 29·6 | 0·67 | 160 | 2·88 |
| 70 | ,, | 87·99 | 84·92 | 79·96 | 26·0 | 34·0 | 30·62 | 42·52 | 21·3 | 9·28 | 1·49 | 15·38 | — 37·5 | 0·53 | 170 | 2·30 |
| 71 | ,, | 87·43 | 84·10 | 77·45 | 30·0 | 36·53 | 35·91 | 47·17 | 17·24 | 7·76 | 1·39 | 15·92 | — 37·8 | 0·86 | 162 | 2·22 |
| 72 | ,, | 87·2 | 85·46 | 79·33 | 25·4 | 29·65 | 29·84 | 37·37 | 30·30 | 8·35 | 1·36 | 11·03 | — 14·0 | 0·73 | 144 | 3·63 |
| 73 | ,, | 86·9 | 82·98 | 78·38 | 29·2 | 32·43 | 35·19 | 41·38 | 25·00 | 7·96 | 1·51 | 12·99 | — 12·9 | 0·58 | 150 | 3·14 |
| 74 | ,, | 86·5 | 84·42 | 78·74 | 26·6 | 33·0 | 31·51 | 37·28 | 24·4 | 8·56 | 1·53 | 12·78 | — 26·2 | 0·67 | 155 | 2·85 |
| 75 | ,, | 82·0 | 84·68 | 79·50 | 30·0 | 36·08 | 35·34 | 45·38 | 27·47 | 6·73 | 1·96 | 8·62 | — 22·1 | 0·77 | 176 | 4·09 |
| 76 | ,, | 87·71 | 83·47 | 77·72 | 28·4 | 32·46 | 34·02 | 41·77 | 18·52 | 9·88 | 2·05 | 16·06 | — 21·4 | 0·58 | 145 | 1·87 |
| 77 | ,, | — | 72·50 | 69·24 | — | — | — | — | 27·78 | 7·00 | 1·28 | — | — | 0·47 | — | — |

| Number. | Method of Clarification. | Difference in Purity (apparent) between Juice and Molasses. | Difference in Purity (real) between Juice and Molasses. | Total Ash. | Ash insoluble in Water. | Ash soluble in Water. | Iron and Calcium Phosphate. | Lime. | Magnesia. | Potash. | Soda. | Silica. | Sulphuric Acid. | Carbonic Acid in the Ash. | Chlorine. | Lime in 100 parts of Ash. |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 61 | Def. | 46·91 | 35·19 | 7·89 | 1·29 | 6·60 | 0·58 | 0 09 | 0·16 | 3·88 | 0·11 | 0·22 | 0·51 | 1·59 | 0·41 | 1·14 |
| 62 | ,, | 49·47 | 44·77 | 8·27 | 2·32 | 5·95 | 0·47 | 0 87 | 0·09 | 3·48 | 0·16 | 0·34 | 0·73 | 1·76 | 0·45 | 10·52 |
| 63 | ,, | 56·58 | 47·08 | 10·16 | 2·61 | 7·55 | 1·06 | 0·60 | 0·22 | 4·44 | 0·14 | 0·21 | 1·08 | 1·99 | 0·30 | 5·91 |
| 64 | ,, | 48·76 | 41·72 | 8·93 | 2·22 | 6·71 | 1·23 | 0·45 | 0·09 | 4·07 | 0·06 | 0·20 | 0·74 | 1·60 | 0·59 | 5·04 |
| 65 | ,, | 48·27 | 42·88 | 9·55 | 2·03 | 7·52 | 0·33 | 0·83 | 0·08 | 4·45 | 0·14 | 0·11 | 0·68 | 1·97 | 0·65 | 8·69 |
| 66 | ,, | — | — | 8·20 | 1 93 | 6·27 | 0·37 | 0·62 | 0·13 | 3·87 | 0·00 | 0·29 | 0·55 | 1·88 | 0·51 | 7·56 |
| 67 | ,, | — | — | 6·90 | 1·78 | 5·12 | 0 58 | 0·60 | 0·10 | 3·20 | 0·08 | 0·03 | 0·32 | 1·71 | 0·17 | 8·99 |
| 68 | ,, | 54·48 | 45·07 | 9·53 | 1·43 | 8·10 | 0·83 | 0·11 | 0·16 | 4·91 | 0·25 | 0·13 | 0·94 | 1·48 | 0·93 | 1·15 |
| 69 | ,, | 49·34 | 37·78 | 8·41 | 0·91 | 7·50 | 0·33 | 0·06 | 0·09 | 3·93 | 0·23 | 0·19 | 1·95 | 1·12 | 0·31 | 0·71 |
| 70 | ,, | 57·37 | 45·47 | 9·28 | 1·71 | 7·57 | 0·40 | 0·54 | 0·17 | 4·67 | 0·51 | 0·08 | 0·76 | 1·85 | 0·89 | 5·82 |
| 71 | ,, | 51·52 | 40·26 | 7·76 | 2·60 | 5·16 | 1·01 | 0·43 | 0·15 | 3·83 | 0·05 | 0·30 | 0·69 | 1·79 | 0·31 | 5·54 |
| 72 | ,, | 56·36 | 49·83 | 8·35 | 1·92 | 6·43 | 0·59 | 0·60 | 0·07 | 3·76 | 0·14 | 0·16 | 1·07 | 1·57 | 0·52 | 7·19 |
| 73 | ,, | 51·71 | 45·52 | 7·96 | 2·02 | 5·94 | 0·45 | 0·63 | 0·12 | 3·36 | 0·14 | 0·14 | 1·05 | 1·51 | 0·45 | 5·65 |
| 74 | ,, | 54·99 | 48·22 | 8·56 | 1·83 | 6·73 | 0·55 | 0·48 | 0·22 | 4·02 | 0·38 | 0·25 | 0·90 | 1·23 | 1·25 | 5·61 |
| 75 | ,, | 46·66 | 38·62 | 6·73 | 2·20 | 4·53 | 1·04 | 0·36 | 0·13 | 2·72 | 0·00 | 0·27 | 0·93 | 1·09 | 0·38 | 5·35 |
| 76 | ,, | 53·69 | 45·94 | 9·88 | 1·99 | 7·89 | 0·96 | 0·48 | 0·00 | 4·88 | 0·06 | 0·16 | 0·70 | 2·27 | 0·37 | 4·86 |
| 77 | ,, | — | — | 7·00 | 2 08 | 4·92 | 0·41 | 0·91 | 0·10 | 3·01 | 0·00 | 0·09 | 0·65 | 1·57 | 0·30 | 13·00 |

Some analyses of final molasses from other countries here follow, and it may be mentioned that such molasses are not generally completely exhausted, but still contain crystallizable sucrose. They are either used for feeding stock, or are fermented and distilled for rum.

FINAL MOLASSES.

| | Trinidad. | Demerara. | Louisiana. | Hawaii. | Antigua. | Peru. |
|---|---|---|---|---|---|---|
| Brix .. .. .. | 79·25 | 80·4 | 80·0 | — | 79·5 | 82·24 |
| Polarization ... | 43 | 45 | 29·4 | 28 | 37·6 | 31·8 |
| Glucose .. .. | — | 8 | 29·4 | 16·65 | 12·9 | — |
| Quotient.. .... | 54 | 56 | 36·8 | — | 47·3 | 38·6 |
| Ash .. .. .. | 4·20 | 7·58 | 9·80 | 12·57 | 6·97 | 8·18 |

The analyses of the Java molasses give rise to the following observations:—

**Degrees Brix.**

As was observed in the case of masse cuites and first molasses, the degrees Brix of the exhausted molasses are always higher than the per cent. of actual dry substance, due to the fact that many of the impurities which accompany the sucrose possess a higher specific gravity than the latter in solutions of equal concentrations. The specific gravity tables corresponding to degrees Brix are calculated for pure sucrose, so that the presence of such impurities causes the degrees Brix (calculated from the specific gravity) to differ from the actual dry substance. The importance of these deviations is shown in the following table, in which the actual concentration, specific gravity, and the corresponding degrees Brix of various substances are brought together:—

| Substances. | Concentration. | Specific Gravity $\frac{17·5}{17·5}$ | Degrees Brix. |
|---|---|---|---|
| Sucrose .. .. .. .. .. .. .. | 10 | 1·04014 | 10·0 |
| Glucose .... .. .. .. .. ... | 10 | 1·04013 | 10·0 |
| Fructose.. .. .. .. .. .. .. | 10 | 1·03990 | 10·0 |
| Sodium chloride .. .. .. ... | 10 | 1·07312 | 17·7 |
| Potassium chloride .... .. .. .. | 10 | 1·06518 | 15·9 |
| Calcium chloride . .. .. .... | 10 | 1·08647 | 20·7 |
| Potassium sulphate ... .. .. .. | 10 | 1·08235 | 19·8 |
| Potassium acetate .. .. .. .. . | 10 | 1·0496 | 12·3 |
| Sodium acetate .. .. .. .. . | 10 | 1·0538 | 13·25 |
| Calcium acetate.. .. . .. .. | 10 | 1·0492 | 12·2 |
| Potassium tartrate .. .. .. . | 10 | 1·0650 | 15·9 |
| Albumen .. .. .. .. .. .. | 10 | 1·0261 | 6·6 |
| Dunder from cane sugar molasses | 9·36 | 1·04356 | 10·8 |
| Sodium glucinate .. .. .. ... | 10·13 | 1·05494 | 13·5 |
| Potassium ,, .... .. .. .. | 9·74 | 1·05358 | 13·2 |
| Calcium ,, .. .. . . .. | 9·92 | 1·04769 | 11·8 |

With a few exceptions, all the substances mentioned here, when in solution, show a higher specific gravity than sucrose and the two other sugars.

Leaving out of question the possible interaction or combination between the different constituents, and assuming that each of these retains its own specific gravity, a solution of the following composition would possess an actual percentage of 70 per cent. dry substance and yet show a density corresponding with 77·7 degrees Brix:—

| | | Brix. |
|---|---|---|
| Sucrose | 40 | = 40 |
| Glucose | 10 | = 10 |
| Fructose | 10 | = 10 |
| Sodium chloride | 10 | = 17·7 |
| | 70 | 77·7 |

All cane molasses contain salts, and since the degrees Brix of solutions of both organic and inorganic salts are in excess of their actual content of dry substance, it is very probable that the degrees Brix of the molasses are too high.

The analyses of a number of artificial mixtures proved that when a solution only contains sucrose and reducing sugars, the sum of degrees Brix and water does not differ much from 100, but falls below 100 when the proportion of reducing sugar increases.

When salts are also present, the sum of degrees Brix and water rises above 100.

| No. | Brix 17·5 C°. | Sucrose. | Reducing Sugar. | Ash. | Water. | Sum of Brix + water. |
|---|---|---|---|---|---|---|
| 1 | 68·1 | 68·12 | 0·0 | 0·00 | 31·9 | 100·02 |
| 2 | 69·2 | 66·8 | 2·4 | 0·02 | 30·7 | 99·9 |
| 3 | 71·7 | 57·5 | 11·0 | 0·03 | 27·81 | 99·51 |
| 4 | 74·7 | 51·6 | 21·7 | 0·03 | 24·85 | 99·55 |
| 5 | 74·8 | 43·8 | 29·4 | 0·03 | 24·7 | 99·50 |
| 6 | 77·9 | 43·6 | 33·3 | 0·03 | 21·27 | 99·17 |
| 7 | 84·3 | 37·4 | 39·2 | 0·03 | 15·0 | 99·30 |
| 8 | 82·4 | 1·8 | 75·0 | 0·06 | 16·6 | 99·0 |
| 9 | 68·6 | — | — | 15·13 | 36·5 | 105·1 |
| 10 | 81·8 | 62·6 | 1·92 | 12·42 | 26·93 | 108·73 |
| 11 | 74·9 | 58·8 | 2·10 | 8·41 | 29·60 | 104·5 |
| 12 | 80·5 | 54·5 | 11·90 | 8·46 | 25·07 | 105·57 |
| 13 | 78·5 | 47·9 | 15·2 | 6·75 | 26·00 | 104·5 |
| 14 | 79·6 | 46·8 | 21·3 | 5·80 | 24·23 | 103·83 |
| 15 | 84·2 | 41·4 | 31·5 | 6·45 | 20·40 | 104·60 |
| 16 | 83·5 | 34·4 | 38·4 | 4·46 | 17·83 | 101·33 |

Although the salts of *inorganic* acids are fully represented in the ash, those of *organic* acids leave carbonates after incineration, the weight of which is only a a part of that of the original salt. In the case of salts containing much organic acid, the amount of ash is smaller than if the majority of the salts had been of inorganic acids. For this reason, as also because every salt in solution has its own relation between specific gravity and dry substance, no fixed factor could be found for correcting degrees Brix to obtain actual dry substance in the molasses. Former researches gave an average factor of 0·6-0·7, so that for an indirect determination of solid matter in an exhausted molasses, the figure for ash was multiplied by 0·6 to 0·7 and the product subtracted from the degrees Brix at 17·5° C. in order to get the actual dry substance. In the analyses recorded in the afore-mentioned tables, the author found an average factor of 0·63, with a maximum of 0·89 and a minimum of 0·47, and only about 20 per cent. of the molasses showing larger deviations from the average than 0·10.

**Polarization.**

It has already been observed that the direct polarization does not yield the true percentages of sugars, because the clarification by means of sub-acetate of lead precipitates part of the fructose and therefore causes the polariscope reading to be somewhat high. But, as has already been remarked, the lack of a suitable clarifying agent, not possessing this or similar inconveniences, compels us to accept this slight error. From the difference between direct polarization and polarization after Clerget, we compute the quantity of other optically active substances in the molasses. The clarification with lead reagent eliminated all the active acids, leaving only the sugars active, and as the sucrose was already accounted for by the polarization after Clerget, the difference between the total polarization and that of the sucrose is due to reducing sugars.

In the tables, this rotatory power (expressed in degrees Ventzke) is calculated on 100 parts of reducing sugar. From these figures we see that the complex of reducing sugars in the molasses derived from carbonatation juices has a rotation of about 0·0; or is so slightly positive or negative that we are justified in the conclusion that the carbonatation process changes the rotation of the reducing sugar in cane juice in such a manner that the individual rotations of the constituents neutralize each other, giving an optically inactive mixture (Compare pages 30 and 157).

The saturation of the excess of lime with carbonic acid, in the carbonatation tanks, stops the action of the lime on the reducing sugars. If subsequent inversion makes the mixture of reducing sugars levo-rotatory, then this rotation persists and will be found in the molasses. But the fact that the rotation of the reducing sugar in molasses is 0·0 proves that the manufacture of white sugar (the chief product of carbonating factories) does not cause an appreciable inversion of sucrose.

The rotatory power of the reducing sugar in juice which is clarified by defecation is not so strongly influenced by the lime as in the carbonatation process; because the amount of lime added is much less, and, moreover, it is at once neutralized by the acids of the juice or by those formed by the decomposition of the glucose. Here, the original composition of the reducing sugar in the juice is the principal factor which influences the rotatory power of these sugars in the molasses; unless, during manufacture, a sourness and inversion of the juice had considerably increased the left-handed rotation. We do not know the original rotation of the reducing sugar of the juice, and therefore cannot tell exactly (either from the percentage, or from the rotation of these constituents) whether a part of the glucose had been formed from sucrose in the course of manufacture or not. But we notice that a strong left-handed rotation occurs in molasses which have been exhausted in one operation (a few hours after the original juice was concentrated to syrup) as also in those molasses which only become exhausted after repeated boiling and crystallizing in tanks for weeks.

We find, for instance, the rotation of the reducing sugar in molasses No. 21 is almost as much to the left as the highest left-handed rotation of one of the molasses from the other list, where the opportunity for inversion has been so much greater.

Taken as a whole, the rotation of the reducing sugars from Table No. I., *i.e.*, the molasses obtained by direct working, was —22·5, and that of Table No. II., of molasses obtained from after-products, only — 19·9, showing clearly that no inversion was produced by the slow work of boiling and cooling of seconds. The inversion of sucrose, mentioned on page 144, must therefore have taken place during evaporation or boiling of the first products.

The Brix being higher than the actual per cent. of dry substance, and the polarization being lower than the actual per cent. of sucrose, we see that two errors co-operate to make the actual purity much higher than the apparent purity. The former is obtained by dividing 100 × the actual per cent. sucrose by the dry substance; and the latter by dividing 100 × the polarization by the degrees Brix.

**Quotient of Purity.**

In the molasses from carbonatation factories, wherein the left-handed rotation of the reducing sugars is almost nil, and where, therefore, one of the sources of error and difference is eliminated, the difference between the actual and apparent purities is not so great as in the molasses from defecated juices. If, therefore, we judge the effect of clarification by the lowering of the apparent purity of the molasses, we shall draw false conclusions as to the value of the carbonatation process. Where the apparent purities show rather large differences between those two systems, the true purities do not differ perceptibly for the various kinds of molasses under consideration, as appears from the subjoined table :—

| | |
|---|---|
| Carbonatation molasses from first sugars (List I.) .. .. .. | 39·9 |
| Defecation molasses from first sugars (List I.) .. . .... | 43·33 |
| Carbonatation molasses from after products (List II.) .. .. | 40·20 |
| Defecation molasses from after products (List II.) .. .... | 40·52 |

These figures do not allow of a rigid comparison, as the purity of the juice was not the same in each case, and in some cases was not known, but the table shows that there is no marked difference in actual purity between these varieties of molasses.

The amount of ash in cane molasses is fairly constant, varying between 7 and 10 per cent, with only slight deviations above or below these limits. The soluble and insoluble parts of the ash are stated apart, and we note that the lime and magnesia were always in the insoluble portion. The soluble ash, which had in every instance an alkaline reaction owing to the presence of alkali carbonates, contained all of the alkali metals, the sulphate, and chlorine.

**Ash.**

The insoluble part was dissolved in hydrochloric acid, evaporated to dryness, dried in a hot air bath; again dissolved in water and hydrochloric acid, and the silica filtered off and determined. The resulting filtrate was saturated with ammonia, and the precipitate filtered off, washed, dried, ignited, and weighed, without further attempt to separate it into its elements. It is recorded in the tables as "iron and calcium phosphate." The quantity of this precipitate is small in the carbonatation molasses, and even in the sample which contains most, the percentage is lower than in defecation molasses. This agrees with the fact already mentioned that the phosphates in clarified juice do not occur in solution but in suspension, and where a filtration of the total juice is possible (as in carbonatation juices) they are more apt to be removed than where nine-tenths of the juice is not filtered at all, but only clarified by subsidation. The same applies to the silica, which is almost absent in the ash of carbonatation molasses, while the defecation molasses contain more silica, which has escaped clarification at the outset.

**Silica.**

**Iron and Calcium Phosphates.**

The silica and phosphates may therefore be considered as accidental impurities in the molasses, because they occurred in suspension in the juice and it was a matter of chance whether they were deposited as scale on the tubes or coils of the evaporation vessels. This is why we did not trouble to analyse these precipitates, which have the same composition as the scale in the evaporating vessels. For the same reason, we did not add the lime contained in these phosphates to the lime which occurs in solution in the molasses as lime salts.

The percentage of lime is expressed on 100 parts of ash in the tables. The average lime content was 15·57 per cent. for carbonatation molasses, and 6·25 per cent. for defecation molasses. In former years, the figures were much higher, and this is not surprising in view of the care now taken to add the proper quantity of lime cream in clarification, and since the usual addition of lime

**Lime.**

cream to the subsided scums and to molasses has gradually been abolished. It is evident that the whole of the lime found in the molasses does not originate from the lime used for clarification, as at least part of that constituent was already present in the raw juice; but the gradual decrease in the percentage of lime is a consequence of the repeated advice not to use more lime in clarification than is strictly necessary. Not because lime is expensive, but because an excess of lime attacks reducing sugar at a high temperature, forming lime salts with their products of decomposition, which are dark coloured, sticky, and easily broken up into acids; this can, of course, be prevented by judicious tempering.

**Magnesia.** The magnesia does not call for particular mention. It is not brought into the juice in the lime (as this is generally pure) but is a normal constituent of cane juice.

**Potash.** The content of potash is of considerable interest. First, we observe that in normal average molasses potash is almost the only alkali metal which is found in the juice. Only in very rare cases does the soda amount to 0·25 per cent. in the ash, but in the great majority of molasses this element was either totally absent or did not exceed a few per cent. of the potash figure. The reason is that even in the presence of a large excess of soda salts in the soil, the cane assimilates potash if available, and if not, it withers and dies rather than assimilate soda salts. The potash content of the molasses varies from a little over 2 per cent. up to 6 per cent.

It appears that the potash content of the molasses or juices depends on the nature of the soil, as is proved by the higher content of potash in molasses made in certain localities, in the present as also in past years.

**Carbonic Acid.** The last column in the tables gives the carbonic acid in the ash, which expresses the quantity of organic acids combined with bases in the molasses and which, after ignition, are transformed into carbonates. The molasses from factories working with carbonatation (by which more of the reducing sugar is transformed into acids) shows a greater content of carbonic acid than the molasses from defecation factories, but this figure is of little further significance.

**Gummy Matter.** The low percentage of gummy matter in carbonatation molasses is very characteristic of the thorough chemical and mechanical clarification, occasioned by the large quantity of lime used in that process. In every instance, the percentage of gums in those molasses is inferior to the lowest figure for that substance in any molasses derived from defecated juice. This is shown in practical working by the fact that syrup from carbonated juice is easily filtered, which is impossible in the case of syrup from defecated juice.

Under "gummy matter" are included all organic substances precipitated by alcohol from an acid solution of the molasses. In many English and American text books the word *gums* is used for organic substance precipitated by basic lead acetate, which is quite another thing.

A sample of the precipitate obtained from an acidulated molasses solution contained 41·8 per cent. of ash. The amount of pentosans, determined by Tollens' method, was 9·13 parts on 100 parts of the organic matter in the precipitate, while the estimation of galactan by the mucic acid method gave a content of 7·06 per cent. of galactan on 100 parts of organic matter. A second sample was found to contain 7·1 per cent. of pentosan on 100 parts of organic matter. The gummy matter does not therefore consist exclusively of pectin, nor of gums dissolved from the fibre, nor yet of slime, but of a mixture of the three in varying proportions.

The difference between total dry substance and the sum of sucrose, reducing sugar, and ash is entered in the tables under the heading "Organic non-sugar."

**Organic Non-Sugar.**

This includes the gum, and the remainder should be divided between organic acids, nitrogenous bodies, caramel, and other products of decomposition of sucrose, glucose, and fructose.

The figure for "organic non-sugar" is rather considerable, and as yet no further subdivision has been made. The carbonic acid in the ash is an approximate measure of the amount of organic acids, but does not yield an absolute value because we do not know what these acids are, nor their combining weights. The acids mentioned in Chapter I. possess the following combining weights:—malic acid = 67, succinic acid = 59, and acetic acid = 60, or on an average = 60. The calculations on pages 154 and 156 showed that on decomposition of glucose by lime, calcium salts of organic acids were formed containing 21·1 and 18 per cent. of calcium oxide respectively, or, on an average, 20 per cent. of calcium oxide, equal to 14·3 per cent. of calcium. The combining weight of those acids is therefore 121, for $\frac{100 \times 20}{(121 - 1) \times 20} = 14{\cdot}3$.

These acids were formed by decomposition with large amounts of lime, and perhaps those formed with smaller quantities possess a still higher combining weight, but as not much of these acids is formed on defecation we obtain a high figure when we assume the average combining weight of all the organic acids to be 100. On incineration only 22 parts of carbonic acid are left, so we may roughly estimate the total amount of organic acid in the molasses to be about 4½ times that of the carbonic acid in the ash. As the figure for carbonic acid is already accounted for, the figure for caramel, nitrogenous bodies and products of decomposition may be found by subtracting 3½ times the carbonic acid from the figure for organic non-sugar, minus the gum.

The nitrogen content of cane molasses does not exceed 0·2 per cent., so that the nitrogenous bodies form not more than 1 per cent. of the weight of molasses. Calculated in this way we find a balance of about 5 per cent. left for caramel and the other products of decomposition, though we must not forget that all errors of analysis are included in that figure, the accuracy of which is not above suspicion.

**Nitrogen.**

**Undetermined.**

| | | |
|---|---|---|
| No. 5.—Organic non-sugar .. .. .. .. .. | | 12·71 |
| Gummy matter .. .. .. .. | 2·39 | |
| Nitrogenous bodies .. .. .... | 1· | |
| Organic acids $3\frac{1}{2}$ $CO_2$ .. .. .. | 3·81 | |
| | —— | 7·20 |
| Balance .. .. .. .. .. .. | | 5·51 |
| No. 17.—Organic non-sugar .. .. .. .. .. | | 11·16 |
| Gummy matter .. .. .. .. | 1·51 | |
| Nitrogenous bodies .. .. .... | 1· | |
| Organic acids $3\frac{1}{2}$ $CO_2$ .. .. .. | 4·16 | |
| | —— | 6·67 |
| Balance .. .. .. .. .. .. | | 4·49 |

**Ratio of Water to Sucrose in Molasses.**

Considering the sucrose and water contents of different molasses, we find little confirmation of the theory that the ratio of water to sucrose fluctuates about the figure 150, being generally much higher.

We must, however, bear in mind that the theory assumes that the combination of sucrose with salts possesses its full proportion of combined water and that the ratio of water to sucrose changes as soon as part of that water is removed by evaporation.

Such a combination will then contain more sucrose on 100 parts of water, than when it possessed its normal water content.

The great difference in the ratio of water to sucrose of various molasses, while their quotients did not vary much, can be ascribed to two diametrically opposite reasons.

The true molasses (with its proper percentage of water and sucrose) may have become diluted by water, and in that case the percentage of sucrose in the water is lower than is required by the composition of the hydrated combination. Or the molasses is concentrated too far and deprived of a part of its combined water, and the ratio is too high. The only criterion which can guide us in this question is the knowledge whether molasses with the low ratios are really saturated or not. If they were diluted, they would be able to dissolve more sugar, and could not deposit crystals when kept at rest.

Among the molasses analysed in these researches, those were selected which had deposited crystals after six months' sojourn in the laboratory, and (as the bottles were stoppered) could be considered as saturated. The amount of crystals was not sufficient to affect the composition of the molasses if they had been removed before analysis. The *quantity*, however, is of no moment here, but only the fact that those molasses deposited crystals, and must have been saturated when analysed.

The molasses not containing grain might either be just saturated, or diluted, and are excluded from the following table; only such samples being taken as were proved to be saturated.

| Number of the Molasses. | Brix. | Dry Substance. | Apparent Purity. | Real Purity. | Sucrose in 100 parts of water. |
|---|---|---|---|---|---|
| 4 | 86·78 | 82·18 | 33·19 | 37·24 | 172 |
| 6 | 86·42 | 80·98 | 39·53 | 42·11 | 179 |
| 7 | 87·39 | 82·48 | 37·53 | 44·28 | 209 |
| 8 | 85·64 | 77·64 | 35·56 | 48·50 | 168 |
| 10 | 91·92 | 85·41 | 33·83 | 41·92 | 244 |
| 13 | 86·42 | 81·15 | 37·26 | 46·58 | 205 |
| 15 | 88·48 | 82·96 | 33·69 | 43·51 | 208 |
| 16 | 87·26 | 82·85 | 26·13 | 36·16 | 175 |
| 17 | 89·37 | 83·94 | 43·86 | 50·56 | 265 |
| 18 | 85·36 | 80·92 | 31·51 | 38·94 | 190 |
| 20 | 90·60 | 85·10 | 32·34 | 42·65 | 244 |
| 21 | 87·38 | 83·61 | 29·75 | 38·64 | 197 |
| 27 | 81·94 | 76·72 | 36·86 | 43·80 | 145 |
| 33 | 89·18 | 84·50 | 39·58 | 48·02 | 264 |
| 35 | 85·04 | 78·40 | 29·29 | 35·59 | 130 |
| 38 | 85·65 | 77·64 | 30·59 | 42·38 | 168 |
| 44 | 82·76 | 78·10 | 32·14 | 39·27 | 140 |
| 50 | 91·16 | 85·30 | 34·48 | 43·61 | 253 |
| 52 | 87·30 | 78·83 | 29·10 | 40·95 | 152 |
| 55 | 83·50 | 78·40 | 29·29 | 35·59 | 133 |
| 60 | 84·40 | 80·06 | 34·60 | 40·12 | 161 |
| 63 | 85·30 | 78·84 | 32·12 | 41·62 | 155 |
| 65 | 88·74 | 84·10 | 36·73 | 42·12 | 223 |
| 68 | 86·83 | 81·50 | 29·02 | 38·43 | 170 |
| 69 | 83·10 | 77·44 | 34·66 | 46·22 | 160 |
| 70 | 84·92 | 79·96 | 30·62 | 42·52 | 170 |
| 71 | 84·10 | 77·45 | 35·91 | 47·17 | 162 |
| 72 | 85·46 | 79·33 | 29·84 | 37·37 | 144 |
| 75 | 84·68 | 79·50 | 35·34 | 45·38 | 176 |

**The low Solubility in the Normal Case.**

Notwithstanding that the molasses were fully saturated, we find less than 150 and more than 250 parts of sucrose on 100 of water, which proves that the low solubility represents a more normal condition than the higher ratios. As stated before, it is evident that the normal combination of sucrose and salts contains from 130 to 160 parts of sucrose per 100 parts of water. When it is lower, the molasses are probably diluted, and when higher the presumption is that the combination

has lost some combined water during the boiling process. It is superfluous to mention that the figures of 130 and 160 are not invariable and that these limits might be exceeded in either direction. The different proportions of sucrose and glucose to salts may vary to such a degree that no fixed values can be stated for any one of them. At any rate, a molasses containing 21·74 per cent. of reducing sugar and a ratio of 339 of sucrose to 100 of water must be considered as over-concentrated with a loss of combined water. This was further confirmed by the appearance of the molasses which set into a semi-solid mass.

**Over-concentration in many Instances.**

This proves that in many factories the evaporation is carried so far that the exhausted molasses lose part of their hydration water and become too thick and viscous. Though crystallization is favoured by a high concentration and this is even indispensable (when the aim is to obtain really exhausted molasses) yet it has the disadvantage of rendering that molasses so stiff that it causes trouble on curing. In many cases, a portion of the hydration water is lost owing to excessive concentration, but may be restored by mixing it in the coolers with diluted molasses, when the hygroscopic compound absorbs water, thereby becoming more fluid and more easily separated on curing, while no sugar is re-dissolved. This explains why, in the experiment referred to on page 248, no sucrose dissolved in the water added, notwithstanding the ratio of water to sucrose being reduced from 264 to 161.

Experience shows that very close boiled masse cuites can be safely diluted to 96° Brix or 90 dry substance, and the mother liquor therein to 85° Brix or 80 per cent. of dry substance, in order to combine the advantages of fluidity and complete exhaustion of the molasses.

The relation between reducing sugars and ash cannot serve as a measure of the sucrose content to be expected in such impure liquids as waste molasses, which still contain in suspension some defecation mud, gums, humic bodies, &c., so that although, generally, a high ratio between reducing sugar and ash coincides with a low sucrose content, unforeseen circumstances may interfere and allow a high ratio to accompany a higher sucrose content in one molasses than in another molasses possessing a lower ratio.

**Definition of a Factory Molasses.**

We have defined an "ideal" molasses as a compound of sucrose, salts, and water in the approximate proportions of 55, 25 and 20, and now define a "factory" molasses as a syrupy compound of sucrose, glucose, and fructose, in varying proportions, in combination with organic and inorganic salts; such compounds being partially deprived of water hydration, and holding in solution or suspension gummy and nitrogenous matter, silica, iron and calcium phosphate and other similar bodies.

## III.—Sucrose Content of and Loss of Sucrose in Molasses.

**Quantity of Molasses depends on the Salt Content of the Clarified Juice.**

The ash content of a normal exhausted cane molasses being fairly constant, the amount of molasses obtained on 100 parts of cane therefore depends on the content of salts in the clarified juice.

After the juice is concentrated and repeatedly crystallized, crystallization ceases as soon as the combination of sucrose with salts is formed to its full amount. All free (that is to say, surplus) sucrose crystallizes out under favourable conditions, and a residue remains which can yield no more sugar, and which contains about 9 per cent. of ash. The quantity of molasses is therefore approximately equal to 11 times the weight of ash in the syrup, and as this ash content varies between very wide limits, the quantity of molasses on 100 parts of cane is equally variable. Canes which yield juice with a high ash content ordinarily yield more molasses than one with a low ash content, as in both cases the ash contents of the molasses is much the same.

It is a remarkable fact that both the ash content and sucrose content of exhausted molasses are generally constant. We saw that the mother-liquors yield sugar crystals until the syrupy combination of sugars and salt is formed, and that it depends upon the ratio of the reducing sugars to salts whether much or little sucrose enters into the combination. A case could occur where the juice contains much salt and little reducing sugar, and consequently the resulting molasses would be rich in sucrose, and could not be reduced to a low purity, although every care and attention be paid to the work. On the other hand, a low ash content might occur with a high content of reducing sugar, and then it is very easy to obtain a very low purity in the molasses.

**The Sucrose Content is also fairly constant.**

**The Purity of Exhausted Molasses is generally about 30.**

Fortunately, under normal conditions, a high ash content is generally accompanied by a high reducing sugar content, so that the ratio between the two is always such that only a little sucrose enters into the combination. The reverse is often met with, *i.e.*, high reducing sugar content and a low ash content, whereas instances of little reducing sugar and much ash are exceptions.

In such cases one will never succeed in obtaining a molasses of low purity, whereas this is easy in the former case.

This accidental coincidence of high ash and high reducing sugar contents causes the mutual relation between sucrose and reducing sugar in normal cane molasses to be rather constant and to show an apparent purity of about 30. But we must not consequently expect to reduce the purity of every molasses to that standard. Exceptional cases may occur of juices containing much ash and only little reducing sugar, and then the molasses contains more sucrose and shows a higher purity than normally.

**Exception to this Rule.**

A few examples of exhausted molasses of high purity follow, and in every case the ash content is high and that of reducing sugars low, causing a high sucrose content.

| Analysis. | 1 | 2 | 3 | 4 |
|---|---|---|---|---|
| Brix | 90·1 | 85·7 | 83·5 | 89·7 |
| Polarization | 37·4 | 38·0 | 35·2 | 35·8 |
| Reducing sugar | 11·9 | 15·4 | 14·5 | 18·87 |
| Apparent purity | 41·5 | 44·3 | 42·6 | 40·0 |
| Ash | 13·26 | 9·33 | 9·3 | 10·86 |
| Potash in the ash | 7·64 | 3·5 | 3·84 | 5·76 |
| Reducing sugar : ash | 0·90 | 1·6 | 1·6 | 1·7 |

On some estates the content of reducing sugar is always low, so that a rather rich molasses must yet be considered exhausted. This is the case on the estate which furnished the molasses Nos. 5, 40, and 64, and where a low ratio between reducing sugar and ash was accompanied by a high purity of the exhausted molasses.

| No. | Ratio of Red. Sugar : Ash. | Actual Purity. | Apparent Purity. |
|---|---|---|---|
| 5 | 1·67 | 46·17 | 35·52 |
| 40 | 1·45 | 44·54 | 38·77 |
| 64 | 1·66 | 48·78 | 41·74 |

Juice from an Egyptian estate contained a great deal of sodium sulphate, and gave the following analytical results:—

| | |
|---|---|
| Brix | 18·2 |
| Polarization | 14·6 |
| Reducing sugar | 0·51 |
| Ash | 0·98 |
| Reducing sugar : Ash | 0·52 |
| Apparent purity | 80·25 |

When such juice is worked up without being mixed with other juice, the purity of the exhausted molasses will not fall below 50, and is similar in character to beet molasses.

This, however, is an extreme case, but when the ratio of reducing sugar to ash amounts to 1·5 it will generally be impossible to reduce the purity of the molasses to 30. The limit of exhaustion lies well above that figure, so that further concentration only renders the molasses more viscous and stiff without causing further crystallization. On this account, it is impossible to reduce the purity of the molasses down to 30 on some estates, and in many cases the juice of one cane variety could be reduced to a molasses of 30 purity, while that of another variety yielded molasses of higher purity even on the same estate. The figures on pages 88, 89, &c., show that one variety assimilates more salts from the soil than another, so it is evident that the nature of some cane varieties may prevent the reduction of purity of the exhausted molasses.

**Ratio of Reducing Sugars to Salts in the Juice.**

An unfavourable ratio of reducing sugar to ash is the only cause which lies beyond the control of the manufacturer; the other causes (to be enumerated hereafter) may be got rid of, so that every juice possessing a favourable ratio of reducing sugar to ash should yield exhausted molasses of 30 apparent purity.

One reason is excessive evaporation, owing to which the cooling mass becomes semi-solid with the formation of a very fine grain, which cannot be recovered but passes through the centrifugal liners along with the molasses. When such a masse cuite is cooled in rotating crystallizers, it can be improved by diluting it to a density of 96° Brix with diluted molasses before curing.* When cooled in fixed tanks, an over-concentrated masse cuite may be improved by covering it with water to a depth of about a foot, allowing it to stand for a month, after which time the supernatant layer of water is removed, and the masse cuite cured.†

**Excessive Evaporation.**

As prevention is better than cure, masse cuites boiled to grain should be diluted at once, and those boiled smooth should not be concentrated too far.

Another reason why masse cuites boiled to string-proof are difficult to cure is that they are cooled too fast, crystals being formed during their transport to the crystallization tanks,‡ so that an excessive quantity of grain is formed which does not grow, and cannot be separated from the molasses. This may be prevented by keeping the temperature of the masse cuite high when it leaves the pan, and by transferring it quickly to the crystallizing tanks.

**Too Rapid Cooling of Smooth-Boiled Masse Cuites.**

These tanks should be neither too small (in order to prevent too rapid cooling) nor too large, but should have a capacity equal to one or two strikes of the pan. If several strikes of the pan are required to fill the tank, the crystallization of the first strike will be disturbed by each subsequent addition of hot masse cuite, which is detrimental to crystallization.

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* See page 248. † Compare page 271. ‡ See page 269.

A high percentage of gums also renders the molasses thick, so that they cannot be properly concentrated. In some cases, the molasses contain so much gummy matter that unless a good deal of water is left in the masse cuite the latter sets hard on cooling. If evaporation be carried too far, crystallization is interfered with, and the crystals formed cannot be separated from the mother-liquor and are consequently lost. If, on the contrary, sufficient water is left in the molasses to keep them fluid, too much sucrose remains dissolved, so that in this case, also, the loss of sucrose is excessive.

**High Gum Content.**

Canes grown in some districts contain much gummy matter, while some varieties of cane are exceptionally rich in this substance. When this is known, a much larger quantity of lime may be used in clarification, and the excess neutralized with sulphurous or carbonic acids.* The gum is thereby precipitated so that the molasses can be exhausted to the usual low purity.

**Repeated Boiling of the same Molasses.**

When the same molasses are repeatedly returned into the masse cuites they become sticky and difficult to separate. The following analyses of a fresh molasses and of one that has been a long time in circulation, both from the same factory, may explain the reason :—

| | Fresh Molasses. | Molasses a long time in circulation. |
|---|---|---|
| Brix | 87·62 | 79·55 |
| Dry substance | 83·51 | 76·47 |
| Polarization | 36·00 | 39·80 |
| Apparent purity | 41·09 | 50·03 |
| True purity | 43·11 | 52·05 |
| Reducing sugar | 27·00 | 20·80 |
| Ash | 5·22 | 4·74 |
| Soluble ash | 2·25 | 2·01 |
| Insoluble ash | 2·97 | 2·73 |
| Silica | 0·16 | 0·19 |
| Iron oxide and alumina | 0·68 | 0·48 |
| Lime | 0·84 | 0·80 |
| Carbonic acid in the ash | 0·75 | 0·59 |
| Gummy matter | 1·94 | 1·90 |

The molasses which have been in circulation a long time contain more water and sucrose than the fresh molasses, due to the addition of "purging water" and solution of the sugar, which in itself is an indication that the molasses were gummy, otherwise purging would not have been necessary. No increase of gums could be detected by analysis, but a much larger amount of dirt settled from the old molasses when solutions of the two samples were kept for some time.

* See pages 132 and 139.

100 grms. of each molasses were dissolved in 100 grms. of water and the solutions allowed to subside in conical glasses. When the deposits ceased to increase in volume they were measured, that yielded by the fresh molasses being 2·4 c.c.; while 11·5 c.c. or 5 times as much was obtained in the other case. The deposit consisted of a slimy substance, containing fragments of incrustations and of decomposition products. It is true that a portion of molasses is withdrawn daily from the course of manufacture and replaced by fresh molasses, but the dirt seems to accumulate in the circulating molasses and renders them so much more sticky than fresh molasses. The remedy is, of course, to reduce the quantity of molasses in circulation* and thus avoid the repeated re-boilings.

**Deficient Clarification.**

If insufficient attention is paid to the subsiding of the syrup or to the steaming and skimming of the molasses, a considerable quantity of a gelatinous substance remains suspended in the molasses, rendering the same very sticky and difficult to separate from the crystals.

The composition of such molasses follows here:—

| | |
|---|---|
| Brix | 85·1 |
| Polarization | 30·8 |
| Purity | 36·2 |
| Reducing sugar | 20·4 |
| Ash | 9·1 |
| Soluble ash | 6·09 |
| Insoluble ash | 3·01 |
| Gummy matter | 2·08 |

The figures are normal and do not afford any clue as to the inferior quality of the molasses. It was, however, very dull in appearance and viscous, while a considerable quantity of dirt deposited from a solution in water. A large quantity of the molasses was diluted with water, and after a few days' rest the supernatant clear solution was decanted off and analysed. The residual dirty liquid was brought to the same density and also analysed.

| | Clear Portion. | Dirty Portion. |
|---|---|---|
| Brix | 65·7 | 66·8 |
| Dry substance | 60·9 | 60·8 |
| Ash | 6·9 | 8·6 |
| Soluble ash | 4·45 | 4·5 |
| Insoluble ash | 2·45 | 4·12 |
| Alkalinity of the ash as CaO | 4·18 | 5·51 |
| Silica | 0·23 | 0·5 |
| Lime | 0·84 | 1·5 |
| Magnesia | 0·15 | 0·16 |
| Sulphuric acid | 0·15 | 0·15 |
| Ironoxide and alumina | 0·42 | 0·72 |
| Gummy matter | 2·40 | 2·21 |
| Viscosity (rate of flow) | 11 min. 15 sec. | 47 min. |

* See page 228.

In the subsided dirty portion we find an increase in silica and organic lime salts, the other constituents being present in much the same proportion in the two liquids. The viscosity, which is here the principal factor, is much greater in the dirty liquid, the rate of flow being four times that of the clear liquid, and it is therefore not astonishing that notwithstanding a normal chemical composition such molasses are very difficult to cure.

It follows that good clarification and subsiding of the juice, combined with careful clarification of syrup and first molasses, can eliminate these gummy substances, so that such inferior molasses do not occur.

**Alleged Occurrence of Dextro-rotating Impurities in Molasses.**

A high quotient of purity of exhausted molasses has been attributed to the presence of a dextro-rotating substance (other than sucrose) in the molasses. In all such cases, a careful examination failed to detect such substances; on the contrary, the direct polarization was always lower than the true sucrose content, owing to the levo-rotating invert sugar present in all cane products. The occurrence of dextro-rotating bodies, other than sucrose, is mentioned in this work on page 141, and it might be thought that these could pass into the molasses and exert their influence there. But it is very probable that the abnormal purities of the juice are not due to a dextro-rotating substance, but to an error in the Brix figure caused by air occluded in the juices. But even if the juice contained a dextro-rotating impurity, this would have been decomposed during concentration to syrup, which latter always showed a normal purity, so that it cannot eventually be found in the molasses.

The absence of dextro-rotating impurities may therefore be considered as settled, and from the foregoing pages it will be seen that there is no reason why the apparent purity of a fully exhausted cane molasses should be much above 30, except in the case of an unfavourable ratio between reducing sugars and ash.

Leaving out of consideration cases where this ratio is 1·5 and thereabouts, we shall only here consider the normal case of an exhausted molasses of 30 purity, or 25 direct polarization.

**Quantity of Molasses for different Purities of Juice.**

The quantity of molasses and the loss of sugar therein depend therefore on the amount of non-sugar in the juice, since an impure juice will yield more molasses than a pure one, and we can easily calculate how much molasses should be obtained on 100 parts of sugar for each degree of purity of the original juice. We assume the polarization of the sugar to be 96·5 and calculate the quantity of molasses of 90° Brix and 30 purity obtained on every 100 parts of such sugar for different purities of the juice. The loss of sugar and other dry

substance in filter press cakes is neglected, and no allowance is made for loss of sucrose by inversion and spilling. The figures between the limits of 77 and 92·9 purity are as follows:—

| | 0 | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 |
|---|---|---|---|---|---|---|---|---|---|---|
| 77 | 46·5 | 46·2 | 45·9 | 45·6 | 45·3 | 45·0 | 44·7 | 44·4 | 44·1 | 43·8 |
| 78 | 43·5 | 43·2 | 42·9 | 42·6 | 42·3 | 42·05 | 41·8 | 41·5 | 41·2 | 40·9 |
| 79 | 40·6 | 40·3 | 40·0 | 39·8 | 39·5 | 39·2 | 38·9 | 38·6 | 38·4 | 38·1 |
| 80 | 37·8 | 37·5 | 37·3 | 37·0 | 36·7 | 36·45 | 36·2 | 35·9 | 35·7 | 35·4 |
| 81 | 35·1 | 34·9 | 34·6 | 34·3 | 34·1 | 33·8 | 33·5 | 33·3 | 33·0 | 32·7 |
| 82 | 32·45 | 32·3 | 32·0 | 31·8 | 31·5 | 31·3 | 31·0 | 30·8 | 30·5 | 30·3 |
| 83 | 29·9 | 29·6 | 29·4 | 29·15 | 28·9 | 28·65 | 28·4 | 28·15 | 27·9 | 27·65 |
| 84 | 27·4 | 27·2 | 26·9 | 26·7 | 26·4 | 26·2 | 26·0 | 25·7 | 25·5 | 25·2 |
| 85 | 25·0 | 24·8 | 24·5 | 24·3 | 24·1 | 23·85 | 23·6 | 23·4 | 23·2 | 22·9 |
| 86 | 22·7 | 22·5 | 22·3 | 22·0 | 21·8 | 21·6 | 21·4 | 21·2 | 20·9 | 20·7 |
| 87 | 20·5 | 20·3 | 20·1 | 19·9 | 19·7 | 19·45 | 19·2 | 19·0 | 18·8 | 18·6 |
| 88 | 18·4 | 18·2 | 18·0 | 17·8 | 17·6 | 17·4 | 17·2 | 17·0 | 16·8 | 16·6 |
| 89 | 16·35 | 16·2 | 16·0 | 15·8 | 15·6 | 15·4 | 15·2 | 15·0 | 14·8 | 14·6 |
| 90 | 14·45 | 14·3 | 14·1 | 13·9 | 13·7 | 13·55 | 13·4 | 13·2 | 13·0 | 12·9 |
| 91 | 12·6 | 12·4 | 12·2 | 12·1 | 11·9 | 11·7 | 11·5 | 11·3 | 11·2 | 11·0 |
| 92 | 10·8 | 10·6 | 10·45 | 10·3 | 10·1 | 9·95 | 9·8 | 9·6 | 9·4 | 9·3 |

At a juice purity of 77, not less than 46·5 parts of exhausted molasses are obtained per 100 parts of sugar, but this is reduced to 9·3 at a juice purity of 92·9.

**Loss of Sugar in Molasses.**

To find the probable loss of sugar in molasses these figures are multiplied by $\frac{90 \times 30}{100 \times 100} = 0{\cdot}27$, and the probable loss on 100 of cane is found by multiplying that product by the yield of sugar per 100 cane, and dividing by 100.

A factory recovered 10·95 parts of sugar per 100 of cane, and the purity of the juice was 85·3. The probable quantity of molasses on 100 cane was, therefore, $\frac{10{\cdot}95 \times 24{\cdot}3}{100} = 2{\cdot}66$ parts, and the loss of sucrose = 0·72; assuming that the purity of the final molasses was 30, the polarization of the sugar = 96·5, and that no sucrose was lost in filter-press mud, by spilling or by inversion.

The statistics of the Mutual Control of Cane Sugar factories in Java show the following losses of sucrose in molasses for different years:—

| Year. | Purity of the Raw Juice. | Sugar made on 100 cane. | Loss of Sucrose in Molasses and unaccounted for. | | |
|---|---|---|---|---|---|
| | | | On 100 Cane. | On 100 Sucrose in Cane. | On 100 Sugar made. |
| 1899 | 88·66 | 11·54 | 1·26 | 9·01 | 10·92 |
| 1900 | 84·18 | 9·85 | 1·32 | 10·76 | 13·40 |
| 1901 | 84·66 | 10·42 | 1·14 | 8·98 | 10·94 |
| 1902 | 84·65 | — | — | — | — |
| 1903 | 84·0 | 10·05 | 1·08 | 8·80 | 10·75 |
| 1904 | 84·69 | 10·91 | 1·05 | 8·05 | 9·62 |
| 1905 | 83·3 | 10·49 | 0·94 | 7·43 | 9·00 |
| 1906 | 83·04 | 10·11 | 1·07 | 8·72 | 10·58 |
| 1907 | 84·61 | 10·91 | 0·95 | 7·25 | 8·71 |
| 1908 | 83·74 | 10·21 | 1·10 | 8·94 | 10·77 |

**Viscosity.**

In discussions regarding the yield obtained from low-grade masse cuites, especially when deficient, too much importance is attached to the viscosity of the molasses, and in most cases this property is made responsible for every inferior yield of sugar from masse cuite. A high quotient of purity of the exhausted molasses is explained away on the assumption that the molasses are so viscous that a high sucrose content cannot be avoided. The author does not believe that viscosity of juices and syrups is sufficient to hinder crystallization, but that careless treatment can cause any molasses to become viscous.

It is a well-known fact that the viscosity of pure sucrose solution increases with the concentration and decreases as the temperature rises, and that this latter decrease is stronger than the former increase.

**Influence of Temperature on Viscosity.**

The same thing is also true of molasses, and the table given below shows how even a slight difference in density of a solution of a given molasses causes a considerable difference in the rate of flow of 200 c.c. from an Engler's viscometer.

Cane molasses: Brix, 83·2; polarization, 25·0; reducing sugar, 30·8; ash, 6·13; diluted to

| Brix. | | Mins. | Secs. |
|---|---|---|---|
| 72·62 | Rate of flow .. .. .. .. .. .. .. .. | 22 | 46 |
| 71·15 | ,, .. .. .. .. .. .. .. .. | 20 | 44 |
| 71·07 | ,, .. .. .. .. .. .. .. .. | 20 | 2 |
| 70·82 | ,, .. .. .. .. .. .. .. .. | 19 | 0 |
| 70·72 | ,, .. .. .. .. .. .. .. .. | 18 | 35 |
| 65·30 | ,, .. .. .. .. .. .. .. .. | 6 | 55 |

Cane molasses: Brix, 80·2; polarization, 31·2; reducing sugar, 26·9; ash, 8·31; diluted to

| Brix. | | Mins. | Secs. |
|---|---|---|---|
| 70·12 | Rate of flow .. .. .. .. .. .. .... | 26 | 44 |
| 69·95 | ,, .... .. .. .. .. .. .. .. | 26 | 11 |
| 69·63 | ,, .. .. .. .. .. .. .. .... | 25 | 45 |

The undiluted molasses was far too stiff to flow from the viscometer, and the experiments were for that reason continued with concentrations of 65-70 per cent. dry substance.

A number of molasses were diluted to about 65 per cent. dry substance, and the rate of flow of those solutions and of sucrose solutions was determined at the temperature of 28° C.

**Viscosity of Various Liquids having the same Density.**

| | Brix. | Dry Substance. | Polarization. | Reducing Sugar. | Ash. | Gummy Matter. | Rate of Flow. | |
|---|---|---|---|---|---|---|---|---|
| | | | | | | | Mins. | Secs. |
| Molasses 1 .. .. | 70·3 | 65·0 | 23·4 | 26·9 | 7·60 | 1·87 | 16 | 30 |
| ,, 2 .... | 69·9 | 65·06 | 26·0 | 30·9 | 6·77 | 1·90 | 20 | 20 |
| ,, 3 .. .. | 69·9 | 64·96 | 25·6 | 24·4 | 7·38 | 0·48 | 22 | 25 |
| ,, 4 .... | 69·0 | 65·24 | 27·3 | 23·5 | 5·37 | 1·80 | 24 | 11 |
| ,, 5 .. . | 70·7 | 65·08 | 20·52 | 25·5 | 7·18 | 1·85 | 18 | 35 |
| Sucrose .. .... | 65·0 | 65·9 | 65·0 | — | — | — | 8 | 57 |
| ,, .. .. . | 70·3 | 70·3 | 70·3 | — | — | — | 24 | 10 |

**Viscosity of Solutions of various Constituents of Molasses.**

These results show that for the same dry substance content, the viscosity of different molasses does not vary much, but is higher than that of sucrose solutions having the same dry substance content. But the non-saccharine impurities of the molasses evidently possess a higher viscosity than sucrose; so in order to ascertain to which constituent this property was chiefly due, the author determined the rate of flow of a number of substances in solution of 65 per cent. at 28° C.

| | Mins. | Secs. |
|---|---|---|
| Sucrose .. .. .. .. .. .. .. .. .. .. .. .. .. .. | 8 | 15 |
| Glucose .. .. .. .. .. .. .. .. .. .. .. .. .. .... | 6 | 33 |
| Fructose .. .. .. .. .. .. .. .. .. .. .. .. .. .. | 4 | 40 |
| Sucrose and Glucose, even parts .. .. .. .. .. .. .... | 7 | 39 |
| Concentrated "dunder" from a cane sugar molasses distillery | 43 | 10 |

The substance which accounts for the increased viscosity is therefore one or other of the complex non-saccharine substances, and not the reducing sugar.

The analysis of the concentrated "dunder," or residue left behind in the still after the distillation of fermented cane molasses, was as follows :—

| | Percent. |
|---|---|
| Dry substance | 61·8 |
| Reducing matter | 3·9 |
| Ash | 15·13 |
| Soluble ash | 3·40 |
| Insoluble ash | 11·73 |
| Carbonic acid in the ash | 2·88 |

It therefore contained some unfermented glucose as well as caramel, decomposition products of sucrose and glucose, and salts. Half of these salts were combined with organic and the other half with inorganic acids.

**Increase of Viscosity by Addition of Impurities.**

10 per cent. of the different substances were then added to a 65 per cent. solution of equal parts of sucrose and glucose, and the viscosity of the mixtures determined at 28° C.

| Substances of which 10 per cent. is added to the 65 per cent. Sucrose and Glucose Solution. | Degrees Brix of the Solution. | Rate of flow Mins. | Secs. |
|---|---|---|---|
| Sodium carbonate | 76·25 | 30 | 23 |
| Sodium chloride | 73·9 | 11 | 40 |
| Ammonium chloride | 65·3 | 5 | 0 |
| Calcium chloride | 76·8 | 27 | 5 |
| Magnesium chloride | 67·7 | 8 | 27 |
| Barium chloride | 77·0 | 9 | 26 |
| Potassium oxalate | 76·3 | 11 | 50 |
| Sodium glucinate* | 70·61 | 16 | 15 |
| Potassium glucinate* | 69·85 | 10 | 6 |
| Calcium glucinate* | 69·45 | 22 | 0 |
| Dry substance from "dunder" | 68·45 | 15 | 40 |
| Gum | 66·9 | 73 | 0 |
| Sucrose and glucose | 67·8 | 46 | 0 |

As stated by Claassen,† sodium salts increase the viscosity more than potassium salts, and again, calcium salts more than sodium salts, as is very clearly shown by the organic salts used here.

With the exception of gum (which, properly speaking, does not belong to the class of constituents examined), sugar is the most viscous. Of the different substances added to a 65 per cent. solution of sucrose and glucose, the viscosity was greatest when the addition consisted of sucrose and glucose.

This seems strange in view of the fact that pure syrups (in which sucrose is chiefly represented) are much less viscous than impure ones, in which the other

* These glucinates are the same products of decomposition of invert sugar by alkalies as has been described in the *International Sugar Journal*, 1908, 284; and on page 309 of this book.

† *Zeitschrift des Vereins für die Rübenzuckerindustrie*, 1898, 535.

**Density the Greatest Factor of the Viscosity.**

constituents are accumulated. But this apparent contradiction is explained when one bears in mind that a pure sucrose solution cannot contain more than 68 per cent. dry substance, while impure molasses can contain even 84 per cent. and over. The greater viscosity of impure molasses is, therefore, due not so much to certain substances having a high viscosity, but to a higher density than a saturated sucrose solution can attain.

As the content of dissolved substances greatly influences the viscosity, it is necessary to examine the viscosity of molasses in an undiluted state, since a comparison of the viscosity of molasses which are diluted to the same dry substance content affords no clue to the viscosity of the undiluted molasses in the practice of sugar manufacture.

As the minute crystals, present in factory molasses, interfere with the viscosity test, the author had recourse to the series of artificial molasses, which had rendered good service in his studies on the formation of molasses.*

A number of mixtures were prepared from sucrose, invert sugar, water, potassium acetate, and calcium chloride (in which the amounts of potash and lime were identical), and allowed to stand until all the sucrose which was liable to crystallize had done so. The saturated mother-liquors were poured off from the crystals, analysed, and tested in an Engler viscometer at 28° C. The figures express the rate of flow of 200 c. c. of the liquids.

**Viscosity of Artificial Molasses.**

Composition of the Mixtures.

| No. | Sucrose. | Invert Sugar. | Salts. | Water. |
|---|---|---|---|---|
| I. | 600 | 0 | 0 | 150 |
| II. | 600 | 15 | 0 | 150 |
| III. | 600 | 60 | 0 | 150 |
| IV. | 600 | 120 | 0 | 150 |
| V. | 600 | 180 | 0 | 150 |
| VI. | 600 | 240 | 0 | 150 |
| VII. | 600 | 300 | 0 | 150 |
| VIII. | 600 | 360 | 0 | 150 |
| IX. | 600 | 420 | 0 | 150 |
| X. | 600 | 15 | 120 | 150 |
| XI. | 600 | 15 | 60 | 150 |
| XII. | 600 | 100 | 100 | 150 |
| XIII. | 600 | 100 | 60 | 150 |
| XIV. | 600 | 220 | 100 | 150 |
| XV. | 600 | 220 | 60 | 150 |
| XVI. | 600 | 440 | 100 | 150 |
| XVII. | 600 | 440 | 60 | 150 |

* *International Sugar Journal*, 1908, 285.

## Results of the Experiments.

| | I. | II. | III. | IV. | V. | VI. | VII. | VIII. | IX. |
|---|---|---|---|---|---|---|---|---|---|
| Specific gravity at 28° C. .. .. | 1·3347 | 1·3413 | 1·3572 | 1·3767 | 1·3771 | 1·3975 | 1·4161 | 1·4244 | 1·4419 |
| Brix at 17·5° C. .. | 68·1 | 69·2 | 71·7 | 74·7 | 74·8 | 77·9 | 80·1 | 81·9 | 84·3 |
| Dry substance .... | 68·1 | 69·3 | 72·19 | 75·15 | 75·3 | 78·73 | 80·87 | 82·5 | 85·0 |
| Polarization .. .. | 68·1 | 66·6 | 56·8 | 50·2 | 41·7 | 41·5 | 37·6 | 36·4 | 35·2 |
| Sucrose.. .. .... | 68·1 | 66·8 | 57·5 | 51·6 | 43·8 | 43·6 | 39·8 | 38·7 | 37·4 |
| Reducing sugar .. | — | 2·4 | 11·0 | 21·7 | 29·4 | 33·3 | 37·1 | 37·8 | 39·2 |
| Ash .. .. .... | — | 0·02 | 0·03 | 0·03 | 0·03 | 0·03 | 0·03 | 0·03 | 0·03 |
| Water .. .. .. | 31·9 | 30·7 | 27·81 | 24·85 | 24·7 | 21·27 | 19·13 | 17·5 | 15·0 |
| Apparent purity.... | 100 | 96·24 | 79·22 | 67·20 | 55·75 | 53·28 | 46·94 | 46·15 | 41·75 |
| Real purity .. .. | 100 | 96·39 | 79·65 | 68·67 | 58·17 | 55·38 | 49·21 | 44·44 | 44·0 |
| Brix + water .... | 100 | 99·9 | 99·51 | 99·55 | 99·50 | 99·17 | 99·23 | 99·4 | 99·3 |
| Sucrose on 100 water | 314 | 218 | 207 | 208 | 178 | 205 | 208 | 221 | 249 |
| Rate of flow .. .. | 14′ 23″ | 16′ 30″ | 36′ | 1° 19′ | 1° 21′ | 4° 2′ | 10° 6′ | 22° 11′ | 118° |

| | X. | XI. | XII. | XIII. | XIV. | XV. | XVI. | XVII. |
|---|---|---|---|---|---|---|---|---|
| Specific gravity at 28°C. .. | 1·4327 | 1·3875 | 1·4153 | 1·4020 | 1·4523 | 1·4011 | 1·4401 | 1·4355 |
| Brix at 17·5°C. .. .. ... | 81·8 | 74·9 | 80·5 | 78·5 | 85·9 | 79·6 | 84·2 | 83·5 |
| Dry substance.. .. .. . | 73·07 | 70·4 | 74·93 | 74·0 | —* | 75·77 | 79·6 | 83·17 |
| Polarization.. .. .. .... | 62·5 | 58·7 | 53·7 | 47·0 | 50·4 | 45·4 | 39·4 | 32·1 |
| Sucrose .. .. .. .. .. | 62·6 | 58·8 | 54·5 | 47·9 | 51·7 | 46·8 | 41·4 | 34·4 |
| Reducing sugar .. .. ... | 1·92 | 2·1 | 11·9 | 15·2 | 20·3 | 21·3 | 31·5 | 38·4 |
| Ash .. .. .. .. .. .. | 12·42 | 8·41 | 8·46 | 6·75 | 8·71 | 5·8 | 6·45 | 4·46 |
| Water .. .. .. .. ... | 26·93 | 29·6 | 25·07 | 26·0 | — | 24·23 | 20·4 | 17·83 |
| Apparent purity .. .. . | 75·17 | 78·36 | 66·71 | 59·88 | 58·67 | 57·16 | 46·79 | 38·44 |
| Real purity.. .. .. .... | 85·67 | 83·37 | 72·73 | 64·73 | — | 60·78 | 52·67 | 41·36 |
| Brix + water .. .. .. . | 108·73 | 104·5 | 105·57 | 104·5 | — | 103·83 | 104·6 | 101·33 |
| Sucrose on 100 water .... | 236 | 198 | 217 | 184 | — | 193 | 203 | 193 |
| Rate of flow .. .. .. .. | 3° 11′ | 0° 33′ | 2° 45′ | 2° 13′ | 46° 30′ | 7° 1′ | 33° 15′ | 66° 4′ |

The viscosity increases with the amount of dry substance, but not in direct proportion, as is shown by the table underneath.

| Dry Substance. | Rate of Flow. | | | Dry Substance. | Rate of Flow. | |
|---|---|---|---|---|---|---|
| | Hours. | Mins. | Secs. | | Hours. | Mins. |
| 68·1 | 0 | 14 | 23 | 75·3 | 1 | 21 |
| 69·3 | 0 | 16 | 30 | 75·77 | 7 | 1 |
| 70·4 | 0 | 33 | 0 | 78·73 | 4 | 2 |
| 72·19 | 0 | 36 | 0 | 79·6 | 33 | 15 |
| 73·07 | 3 | 11 | 0 | 80·87 | 10 | 6 |
| 74·0 | 2 | 13 | 0 | 82·5 | 22 | 11 |
| 74·93 | 2 | 45 | 0 | 83·17 | 66 | 4 |
| 75·15 | 1 | 19 | 0 | 85·0 | 118 | 0 |

* The figure for dry substance was lost during analysis.

It is a peculiar fact that for a given dry substance content the saturated solutions containing salts have a greater viscosity than those which only contain sugars, while in the table for unsaturated solutions given a few pages back the solution free from salt was more viscous than one in which the amount of salt was replaced by sugars. It appears that, at high concentrations, the viscosity of the complex of sugars and salts differs much from that at low concentrations. At a concentration of 65 per cent. dry substance, the non-sugar in the molasses is much more viscous than that of a sucrose solution of the same concentration, whereas when 10 per cent. of dry non-sugar is added to a 65 per cent. sucrose-glucose solution, a much less viscous mixture results than when 10 per cent. of the same mixture of sucrose and glucose is dissolved. This is another warning against judging the viscosity of the molasses from their behaviour in a diluted state, instead of determining their viscosity in their original, saturated, but crystal-free condition.

In the case of artificial molasses, some regularity was detected in the increase of viscosity with an increase of dry substance, but such regularity is not so obvious in practical working. There, we sometimes encounter very high viscosities combined with comparatively small dry-substance contents, and this is due to the presence of other bodies in the molasses than were made use of in the artificial liquids.

**Influence of Gums and Suspended Matter.**

The most prominent among these are gummy substances, decomposition products of reducing sugar, and also perhaps silica. It is a well-known fact that cane-gum and pectin can sometimes occur in exhausted molasses to an extent of 5 or 6 per cent., and it is very evident that such bodies might increase the natural viscosity of molasses to a considerable extent.

The decomposition products of reducing sugar also affect the viscosity of syrups and molasses. The quantity of silica found in molasses (not exceeding 0·20-0·30 per cent.) is too small to be capable of increasing its viscosity.

To ascertain the influence of gum, so much cane-gum was mixed with a sucrose solution of 66·5 per cent. that its content amounted to 3·86 per cent. on 100 parts of the mixture. Dry glucose was added to a second portion of that solution so that the dry substance content was equal to that of the gum-containing one. The two liquids then contained the same amount of dry substance, sucrose, ash, &c., but as in one case 3·86 per cent. of the glucose was replaced by a similar weight of gum, any difference in viscosity was exclusively due to the gum. The rate of flow of the gum-containing molasses was 31 minutes and that of the other 10 minutes 40 seconds, showing the great influence of gum on the viscosity. An addition of 0·50 per cent. of dissolved silica had no influence, while the addition of a heated calcium glucinate solution could not be ascertained because that

substance separated black, greasy flocks, which choked up the apparatus and thus prevented the determination.

A further cause of viscosity of molasses is due to the presence of finely divided suspended matter. This was proved by the following experiments in which three portions of similar sucrose solutions were triturated with 5 per cent. of finely pulverized sugar, 2 per cent. of fine river silt, and 2 per cent. of washed and dried filter-press mud respectively. The rate of flow of the four liquids was as follows :—

| | Mins. | Secs. |
|---|---|---|
| Sucrose solution without addition .. .. .. .. .. .. | 13 | 10 |
| ,, ,, with 5 per cent. pulverized sugar .... | 21 | 0 |
| ,, ,, ,, 2 per cent. river silt.. .. .. .. | 18 | 35 |
| ,, ,, ,, 2 per cent. dried mud.. .. .... | 21 | 0 |

This shows how finely divided insoluble substances, such as often occur in molasses, greatly influence the viscosity and can cause large differences in that property when the dissolved constituents are normal.

Apart from these differences, due to suspended matter, we are justified in declaring that the viscosity increases with the dry substance content, and that a molasses, which has been over-concentrated, has so increased in viscosity that much labour and trouble are required to separate it from the crystals during curing operations.

**Influence of Temperature.**

One factor has so far remained unconsidered, viz., temperature. All the determinations of viscosity recorded here are made at 28° C. and relate exclusively to that temperature. As Claassen has proved, temperature has a great influence on viscosity, especially between 15° and 40° C. In order to investigate this influence three molasses (free from minute sugar crystals) were tested at different temperatures with these results:—

| | I. | II. | III. |
|---|---|---|---|
| Dry substance .. .. | 69·73 .... | 72·73 .... | 80·50 |
| Polarization .. .... | 35·4 .... | 28·0 .... | 22·4 |
| Gum .. .. .. .. | 5·94 .... | 1·72 .... | 1·35 |

| | Hrs. | Mins. | Secs. | Hrs. | Mins. | Secs. | Hrs. | Mins. |
|---|---|---|---|---|---|---|---|---|
| Rate of flow 28° C. .. .. | 3 | 50 | 0 .. | 3 | 16 | 0 .. | 27 | 0 |
| ,, 35° C... .... | 1 | 42 | 0 .. | 1 | 47 | 0 . | 19 | 40 |
| ,, 40° C. .. .. | 0 | 44 | 10 .. | 0 | 53 | 10 .. | 6 | 43 |
| ,, 45° C... .... | 0 | 29 | 10 .. | 0 | 36 | 45 .. | 4 | 13 |
| ,, 50° C. .. .. | 0 | 20 | 0 .. | 0 | 25 | 10 .. | 2 | 36 |
| ,, 60° C... .... | 0 | 12 | 40 . | 0 | 11 | 40 .. | 1 | 30 |

We see that temperature has a great influence, especially temperatures under 45° C.; above this point, a decrease in temperature is not so evidently accompanied by an increase in viscosity.

These researches have taught us to regard concentration and temperature as the principal factors of viscosity; and as secondary ones, the content of gum, the decomposition products of reducing sugar, and other deposits, and the presence of fine grains of sugar. It is therefore desirable from a manufacturer's point of view that the molasses be as little viscous as possible in order to enable it to be easily separated from the crystals without much washing in the centrifugals. This may be accomplished by taking care not to push the concentration of the last masse cuites too far; or, in case they have become too much concentrated, to dilute them with dilute molasses in a judicious manner until the compounds of sucrose with salts have absorbed their full proportion of hydration water. Not to exceed that point (in which case loss would ensue by the dissolving of sugar in the excess of water) the dilution should not be conducted so far, but be stopped just before this hygroscopic combination is fully saturated. A water content of 10 per cent. (equivalent to 96° Brix) for masse cuites (boiled to grain) after dilution, and a water content of 20 per cent. (equivalent to 85° Brix) in the resulting molasses, are the most favourable.

The cooling must not be pushed further than 45° C. and should remain preferably a couple of degrees over that figure, as at that temperature crystallization is finished, and below that the viscosity increases so considerably that cooling down to that point only causes trouble without any compensating advantage.

The increase of viscosity occasioned by secondary circumstances, such as gummy matter, insoluble substances or false grain, can, of course, be avoided by well conducted clarification, subsiding, filtering, boiling, and cooling processes. Thus we see that a high viscosity is by no means a property of some particular juice or syrup; but, on the contrary, it is within the power of any sugar boiler to keep his molasses fluid and capable of easy separation from the sugar crystals.

**Conclusions.** The final conclusion drawn from these considerations is that, provided the manufacture is well conducted, the only reason for obtaining an exhausted molasses of over 30 apparent purity is a ratio of reducing sugar to ash below 1·5.

This causes more sucrose to enter into saline combinations than corresponds with a purity of 30, and as there is no remedy, we are obliged to submit to it. All other causes, such as gummy matter, decomposition products of sucrose or reducing sugar, defective clarification of juice, syrup, or molasses, excessive concentration of masse cuites either in the pan or in the coolers, cooling to too low a temperature, false grain, &c., are capable of improvement or may be avoided.

# IV.—Utilization of the Molasses.

## 1.—Recovery of Sugar from Molasses.

**Extraction of Sucrose from Molasses.**

Since about 8 per cent. of the sugar present in the cane is lost in molasses, it is not surprising that attempts have been made to extract this portion also.

As explained in the foregoing pages, the sucrose in molasses is uncrystallizable because it is associated with salts in combinations which are stable in a concentrated state, but unstable and even dissociated to a considerable extent in a diluted one. The sucrose can only be recovered by some process which removes the salts from the diluted solution and leaves the sucrose free, so that on concentration it can be induced to crystallize. This can be performed by diffusion

**Osmosis.**

through a membrane of parchment paper which is in contact with the diluted molasses on one side and with water on the other. As salts diffuse more rapidly than sugar, the molasses contain less salts after osmosis than before, and this process has, therefore, found some application in the beet-sugar industry and the refinery.

Cane molasses were subjected to prolonged osmosis in a small apparatus, and the effect is shown by the following analyses wherein the figures are calculated on 100 parts of dry substance:—

| | Before Osmosis. | | After Osmosis. | | Waste Water. | |
|---|---|---|---|---|---|---|
| | Original. | On 100 Dry Substance. | Original. | On 100 Dry Substance. | Original. | On 100 Dry Substance. |
| Brix .. .. .. .. .. | 85·1 | — | 40·5 | — | 25·3 | — |
| Sucrose .. .. .. .. | 29·4 | 38·2 | 15·5 | 41·3 | 7·2 | 31·7 |
| Reducing Sugars .. .. | 28·2 | 36·7 | 14·8 | 39·5 | 7·01 | 30·9 |
| Ash .. .. .. .. .. | 6·88 | 8·93 | 2·9 | 7·73 | 4·17 | 18·3 |
| Water .. .. .. .. .. | 22·98 | — | 62·48 | — | 77·3 | — |
| Apparent purity .. .... | 34·5 | 34·5 | 38·3 | 38·3 | — | — |
| Glucose ratio .. .. .. | 96 | 96 | 95·5 | 95·5 | 97 | 97 |
| Total sugars : Ash .... | 8·4 | 8·4 | 10·5 | 10·5 | 3·4 | 3·4 |

The sucrose and reducing sugar have diffused in much the same proportion as that in which they originally occurred, but the salts have diffused to a much larger degree. From the ratios between the various constituents in the three products we calculate that 11·5 per cent. of the dry substance has diffused from the molasses, leaving 88·5 per cent. Since more salts have diffused than sugars, it is evident that the osmosed molasses do not contain sufficient salts to combine with all of the sugar, so that after concentration some sugar will crystallize out. At the same time, so much sucrose and reducing sugar will go to waste that the profit of the osmosis process cannot be considerable. Further, this process requires much dilution and therefore a large amount of fuel, so that the cost of recovering a small quantity of sugar from the molasses is scarcely profitable.

**Steffen's Extraction Process.**

Experiments with Steffen's extraction process and similar methods, which had proved successful with beet molasses, were unsuccessful in the case of cane molasses, because of the high content of reducing sugar and the requirement of low temperatures.

**Extraction Processes chiefly based on Excise Regulations.**

The profit obtained by extracting sugar from molasses in European countries is not due to the value of the sugar but chiefly to the difference in duty levied on sugar from the roots and from the molasses. As soon as the excise regulations were changed in such countries, so that sugar from molasses paid the same duty as that from roots, the osmosis process was abandoned.

## 2.—Use of Molasses as Fodder.

Exhausted cane molasses are readily consumed by horses, mules, oxen, and sheep, but the great difficulty is to store and transport the viscous fluid and to divide it into proper rations. An important step was made by Hughes, who found that a mixture of fine bagasse and molasses formed a dry meal, having a high sugar content, and suitable for storage and transport in bags without sweating or staining the packing material.

**"Molascuit."**

This product, named "Molascuit," consists of a mixture of 75 parts of molasses and 25 parts of dry, fine parenchyma fibre (pith) of sugar cane. It should not contain less than 45 per cent. of total sugars (sucrose and reducing sugars) and not more than 15 per cent. of moisture. The mixture should be homogeneous and dry, so that the bags in which it is transported do not sweat nor become stained, while the molascuit itself should not become sticky, nor cake.

**Preparation.**

The principal condition is that the fibre be exclusively from the pith-cells and not from the fibro-vascular bundles, because, as we showed on page 87, the absorptive power of the latter is so much inferior to that of the former. Further, the fibre from the parenchyma or pith cells is much more digestible than that of the bundles, which moreover exert an irritating action on

the intestines. The fine bagasse (chiefly of the fibre of the pith) can be obtained by sifting the green bagasse as it comes from the mills. Large sieves of coarse gauze are placed under the slide of the mills, the bagasse falls on them, and the finer parts pass through. This fine bagasse is dried to 90 per cent. of dry substance either by hot air, or by steam, or on the chimney flue, after which it is sifted again, if necessary, and mixed with hot concentrated molasses of 88° Brix (85 per cent. of dry substance).

### 3.—Use of Molasses as Fuel.

**Calorific Value.** As molasses contains about 70 per cent. of organic substances, many attempts have been made to use it as fuel.

The calorific value of molasses containing 9·28 per cent. ash, and 19·4 per cent. moisture, was found to be 5515 B.T.U., or (calculated to 100 parts of dry organic matter) 7366·4. It is, therefore, superior to that of sucrose (7119), and equal to 80 per cent. of that of cane fibre (8550).

The use of molasses as fuel presents certain difficulties; the molasses burns very badly without addition of some other fuel, and, when mixed with bagasse,

**Mixing with Bagasse.** forms a voluminous mass of coke, which chokes up the grate and hinders the free access of air. At an elevated temperature the lime and alkali of the molasses combine with the silica from the bagasse, forming a kind of glass, which covers the grate bars and again prevents the admission of air.

In some factories, where the bagasse was sprinkled with molasses on its passage from the mills to the furnaces, much labour was necessary to clear the furnace and remove the slag from the grate, so that in every instance the addition of molasses to the bagasse was discontinued.

In very large factories it will pay to build special molasses furnaces, such as are used in several cane-growing countries, and were described in the *Hawaiian Planters'*

**Special Molasses Furnaces.** *Monthly* of December, 1903. These furnaces have no grate, and the air enters under the fuel through holes made in the walls. The molasses are either blown into the furnace by means of a steam jet through a kind of injector, or are poured upon an iron plate in front of the furnace, allowed to dry, and thence shovelled into the fire. This is started with a little firewood, but, once being lit, it is kept burning with the dried molasses. The combustion is perfect, the ash fine, and almost free from carbon, while the heating power of two parts of molasses is approximately equal to one part of dry firewood. In most cases, the amount of available molasses will be insufficient to furnish fuel for one boiler, so that only very large factories can profitably adopt this practice.

The ash is very rich in potash, containing as much as 35 per cent. of this constituent. It may therefore be used by glassmakers, and, after being refined, by soapmakers, or as a fertilizer.

### 4.—Molasses as Raw Material for Rum.

The principal application of waste molasses is in the manufacture of rum or arrack in countries where the excise regulations permit of this, and where there is a market for the distilled product.

As this industry is quite distinct from sugar making, we shall not enter into a discussion of fermenting and distilling here, although these form a very important item in many sugar factories.

### 5.—Use as a Fertilizer.

Some estates employ molasses as a fertilizer, or as a means to break up rocky subsoil, thus facilitating tillage. When used as manure, molasses are simply poured over the earth on the banks.

All attempts to manufacture gas from molasses have failed, nor can molasses be used for the manufacture of cyanides and other nitrogenous substances as is the case with beetroot molasses, because cane molasses only contain trifling amounts of nitrogen.

The question how to utilize molasses in countries where the transport of molascuit, rum, &c., to other countries is too expensive is, therefore, still unsolved, and generally the only way of getting rid of this troublesome by-product is to throw it into the nearest stream.

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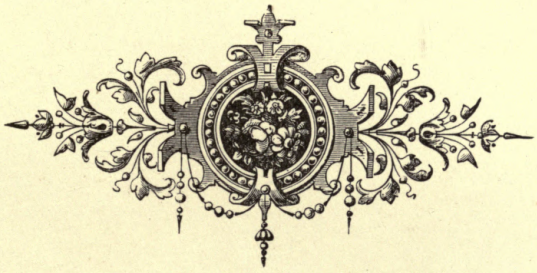

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